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Part II

EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

MECHANICAL PROPERTIES OF AL-AL_2O_3 COMPOSITES

Part II : FRITTOXAL

by

D.J. BOERMAN, M. GRIN and M. VEAUX

1969

Joint Nuclear Research Center
Ispra Establishment - Italy

Metallurgy and Ceramics Department

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ABSTRACT

This report, based in part on already published data, deals with the main mechanical properties of Frittoxal which is the trade name of an aluminium-alumina composite produced by the Trefileries et Laminoirs du Havre (now Tréfimétaux, Paris, France). Frittoxal is a product which differs from the $Al\text{-}Al_2O_3$ composites, known as SAP, mainly by the fact that the starting material is not really a powder of round particles but a powder composed of aluminium flakes.

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KEYWORDS

MECHANICAL PROPERTIES
ALUMINUM
ALUMINUM OXIDES
POWDERS
FABRICATION
HEAT TREATMENTS

CONTENTS

MECHANICAL PROPERTIES OF $AL-AL_2O_3$ COMPOSITES
PART II : FRITTOXAL

Introduction (*)

This is the second part of a general report on mechanical properties of $Al-Al_2O_3$ composites. It is based mainly on data already published in the literature. It deals with the main properties of Frittoxal which is the trade name of an aluminium-alumina composite produced by the Tréfileries et Laminoirs du Havre, (now Tréfimétaux, Paris, France). This Company owns the Frittoxal patents (Ref. 67 and 68).

Frittoxal was considered as a possible solution for the fuel element sheaths (temperature 450°C) and the pressure tubes (temperature 450°C and pressure 20 atm) for the ORGEL project.
A research programme under Euratom contract was therefore carried out at the TLH between 1960 and 1964 in close cooperation with the Metallurgy and Ceramics Division of the European Joint Research Centre at Ispra. This programme was headed by Messrs. J. Herenguel and P. Jehenson for the TLH and Euratom respectively (Ref. 71-85).

Frittoxal is a product which differs from the composites $Al-Al_2O_3$, known as SAP products, mainly by the fact that the starting material is not really a powder of round particles but a powder composed of aluminium flakes.

(*) Manuscript received on 29 November 1968.

INDEX OF DIAGRAMS AND TABLES (1)

Type of test	Diagrams of mechanical properties vs. temperature					Diagrams vs. Flake thickness		Diagrams vs. Oxide thickness	
	A-120-20-D	A-50-10-D	A-40-15-D	A-20-07-D	A-10-01-D	Room temp.	High temp.	Room temp.	High temp.
Tensile	8 - 9 18 - 19 - 20 4 - 15	10 - 11 18 - 19 - 20 4 - 11 - 15	12 - 13 18 - 19 - 20	14 - 15 18 - 19 - 20 3 - 11 - 15	16 - 17 18 - 19 - 20 3	28 - 29	28 - 29	31	31
Creep	24 - 25 5 - 8	5 - 8 - 12	7 - 8	26 8 - 12	8		30		32
Burst tests on finned tubes				9	9				

(1) The underlined numbers refer to table numbers and the others to diagram numbers.

1. MANUFACTURING PROCESS (Ref. 69, 79 and 85)

The manufacturing process for the fabrication of semi-finished products is summarized in the flow sheet in fig. 1.

1.1 Preparation of the Powder

The starting material is a thin aluminium sheet (about 10 μ) which is milled in a stamp-mill in the presence of stearic acid as a lubricant; this particular type of milling is characterized by:

- breaking up of the initial sheet
- a reduction in thickness,

which leads to a powder made up of flakes whose dimensions (length and thickness) depend on the milling time. The powder is then sized in two stages:

a) in a first step the powder is sized according to its greatest dimension on mechanical sieves;

b) in a second step the powder is sized according to its thickness in pneumatic graders.

An idea of the distribution of the thickness in a batch is given by fig. 2 and the following table.

Nominal dimension	Distribution
18 μ	85% of particles between 25 and 10 μ
8 μ	85% of particles between 12 and 4 μ
4 μ	85% of particles between 6 and 2 μ

In fact, the average thickness is a fictitious value determined on the basis of the surface of water covered by a certain amount of powder whose particles are set as close together as possible ("covering power"). The thickness "e" in microns is obtained from the formula:

$$e = \frac{3410}{P} \qquad \text{(from Ref. 69)}$$

where P is expressed in g/cm^2

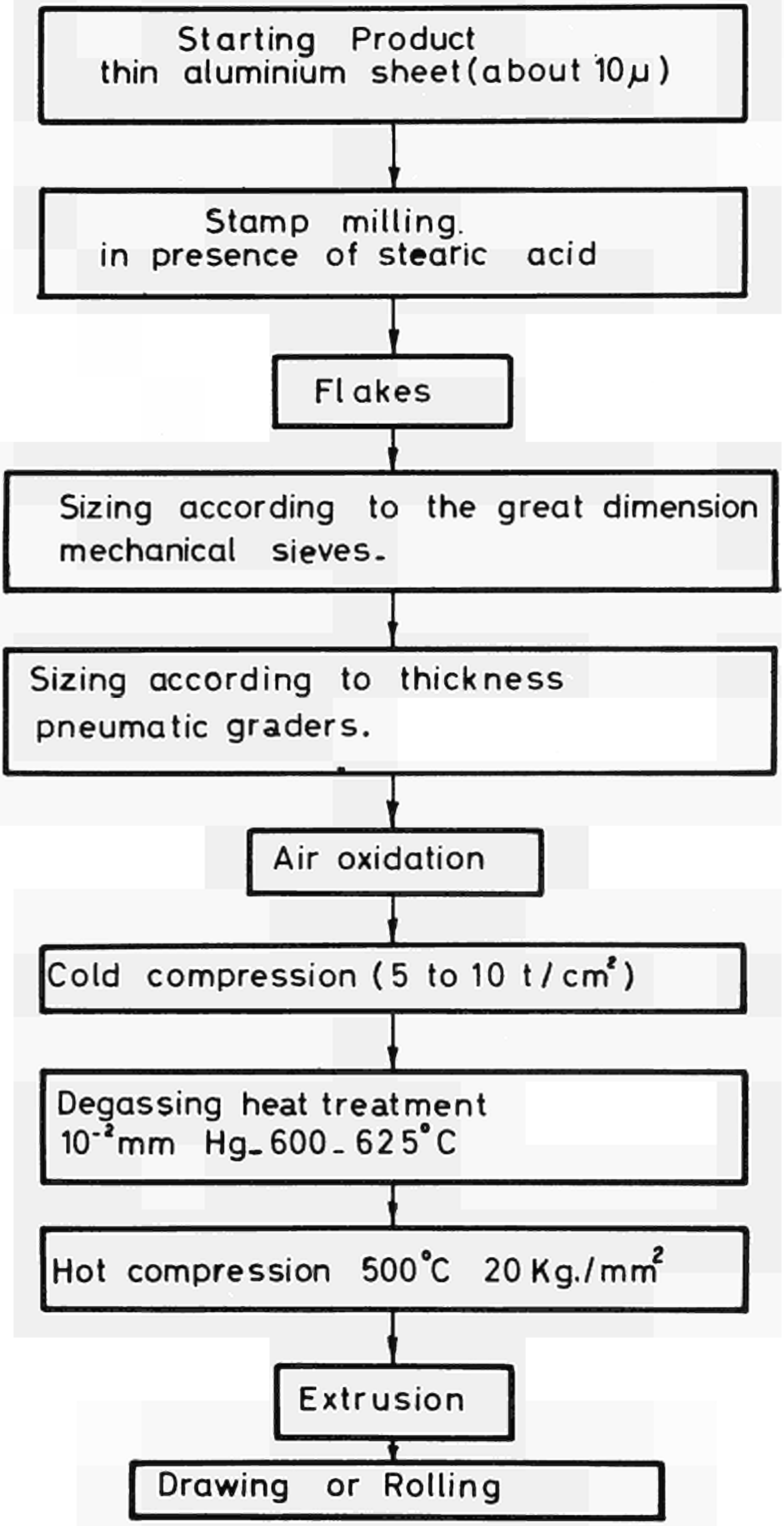

Fig.1 Manufacturing process for the production of finished products in Frittoxal.

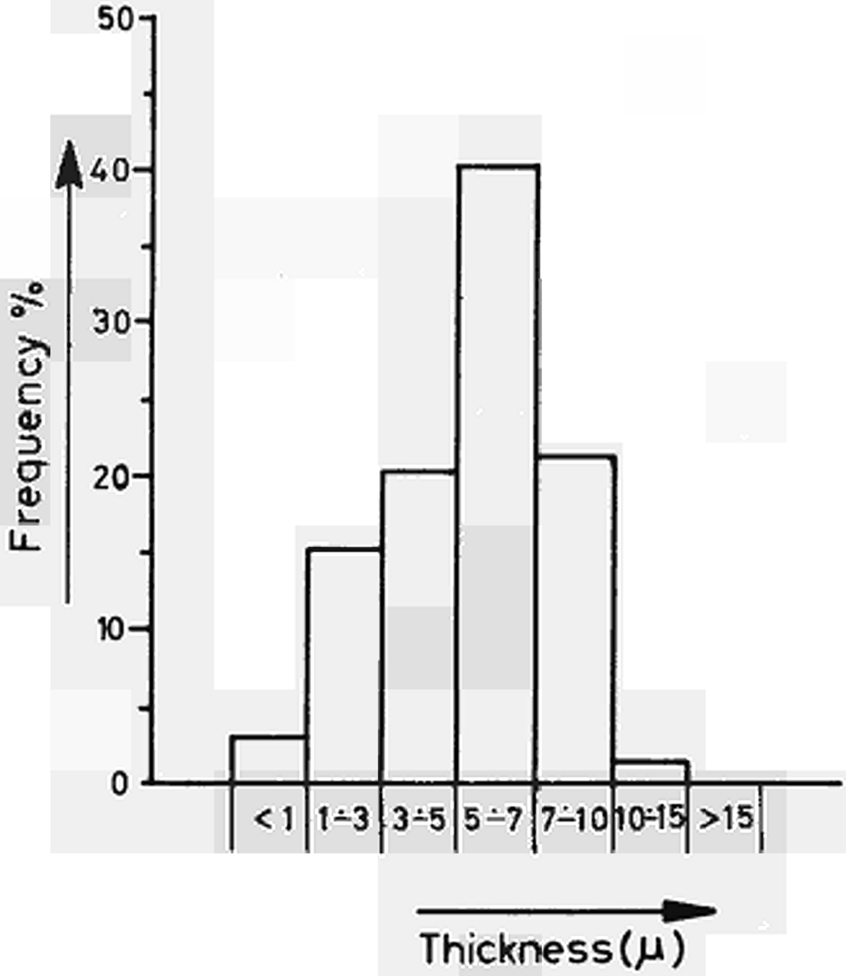

Fig. 2 Distribution of thicknesses for powder of nominal thickness 5μ (from Ref. 85)

1.2 Oxidation Process

After the milling process the aluminium particles are coated with a thin film of aluminium stearate. By heating in air at a temperature higher than 450°C the powder is sufficiently degreased to permit sintering without any previous chemical cleaning.
Oxidation of the particles is achieved, in an electrical rotary furnace, by controlled heating in a mixture of oxygen and nitrogen; the thickness of the alumina film is adjusted by varying the time, the temperature and the oxygen content.

The powder is then subjected to sifting (mechanical sieve) and, possibly, to a final sizing in order to eliminate coarser aggregates of particles (generally with a high alumina content).
An example of the oxidized powder is given in the micrograph in fig. 3.

Degree of oxidation

After the oxidation process the degree of oxidation of each batch is determined by chemical analysis; the oxide film can be estimated on the basis of the alumina content provided that the total surface and the density of the oxide are known.

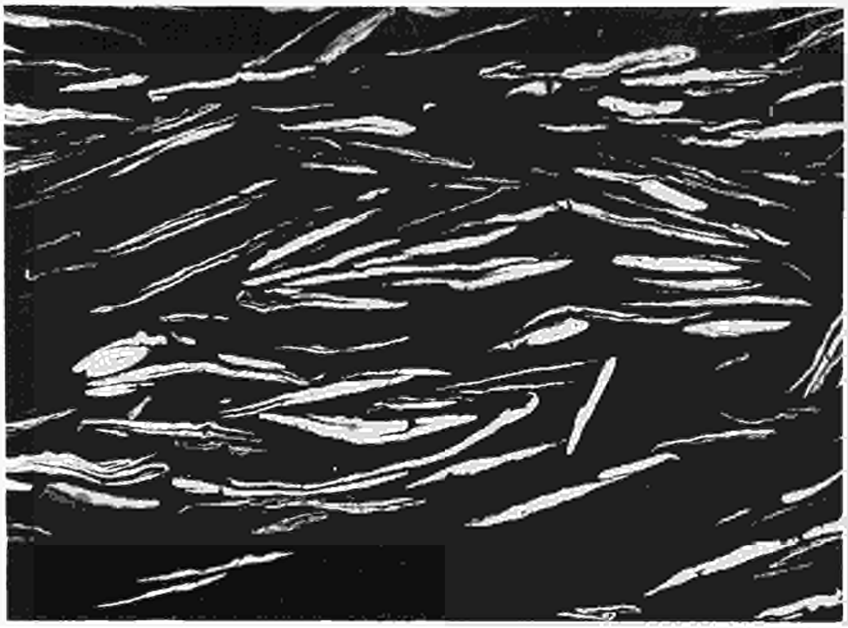

Fig. 3: Cross-section of oxidized particles
Grade A-50-10 magnification 130 (Ref. 79)

For this last value it is assumed that the alumina is one with a density of 4.0. In the case of flakes the surface is evaluated from the value of the "covering power".
We obtain (Ref. 69):

$$f = a.e.\ (0.00337)$$

f = thickness of the oxide film in μ

a = alumina content (weight %)

e = average thickness of the flakes in μ

Identification of the Powder

Each powder is identified by the mean thickness of the particles and of the oxide film.

1st conventional sign: indicates the type of the starting aluminium
- "A" for an aluminium of 99% purity (A4)(Ref. 77) (Fe + Si + Mn + Cu + Zn $\leq$ 1%)
- "A 9" for an aluminium of 99.9% purity (A9) (Fe + Si $\leq$ 0.05%)

1st figure: average thickness of the particles (in μ) multiplied by 10;

2nd figure: average thickness of the oxide film (in μ) multiplied by 100.

For example, the denomination A-120-20 describes a powder composed of flakes 12 μ thick with an oxide film of 0.2 μ manufactured from an aluminium of 99% purity.
The batches of powders are then well defined, either for the particles or the oxides thickness, these two factors being of major importance as far as the mechanical properties of the finished products are concerned; they are much more significant than the oxide content, specially when the thicknesses of the particles vary within broad limits.

1.3 Cold Compression

This compression, with a pressure of 5 to 10 t/cm^2, leads to a compact product with an "open" structure.

1.4 Vacuum Treatment (Ref. 68 and 69)

A special degassing thermal treatment at a temperature of 600-625°C under 10^{-2} mm Hg vacuum gives some improvements on the quality of the semi-finished product, the lowering of the gas content being of fundamental importance with regard to the high temperature stability and the welding properties.

1.5 Hot Compression

After the vacuum treatment the billet, in a tight container, is compressed at a temperature of about 500°C under a pressure of 20 kg/mm². The quality of the sintered product depends closely on the conditions of this last compression (time, temperature, pressure), which in turn depend on the starting product.

The density of the sintered product is a good indication of the quality. As an example fig. 4 shows the curve for the density versus the compression temperature of a product manufactured from powders of particles 10 µ thick.

It is clearly seen that there is a critical temperature beyond which the density becomes constant (2.743 in our example).

1.6 Extrusion and Drawing or Rolling (Ref. 71, 72 and 79)

The solid billet (diameter 45 mm) in the case of rods, or a hollow one for the fabrication of tubes, is then extruded in a vertical press (power 150 tons) at a temperature of about 500°C.

The main conditions concerning the fabrication of finished products of various shapes are summarized below; it must be considered that the conditions of fabrication described are concerning a pilot production of laboratory scale; conditions for industrial scale could be slightly different:

1.6.1 Fabrication of Smooth Tubes (11.6/10 mm)

The billet is drilled to a diameter of 10.5 mm. Extrusion ratio 36 (from 45/10.5 mm to 12/10.2 mm).

Annealing 1 hr at 600°C

Pickling

Drawing at room temperature from 12/10.2 to 11.6/10 (reduction of area 18%)

Annealing 1 hr at 600°C

Pickling

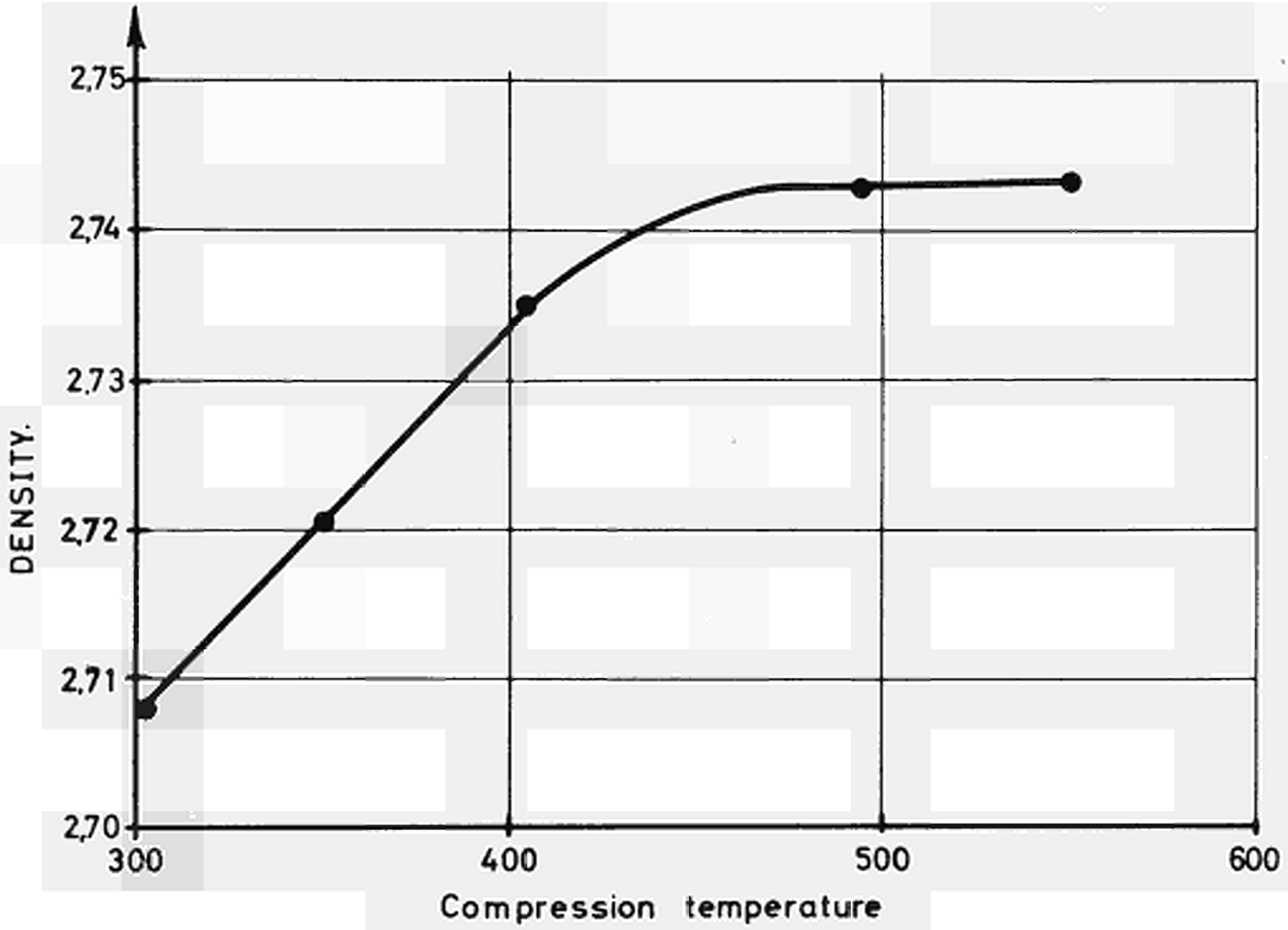

FIG. 4 Flakes 10μ thickness. Density versus compression temperature
(Pression 90Kg./mm², time 2 min)
Alumine content. 6,6 %
(from Ref. 69)

1.6.2 Fabrication of Rods (diameter 10.5 mm)

Extrusion (45 to 12 mm)

Annealing 1 hr at 600°C

Pickling

Drawing to 10.5 mm

Annealing and pickling

1.6.3 Fabrication of Ribbons (e = 0.5 mm)

Extrusion from diameter 45 mm to 12 x 6.5 mm

Hot rolling from 6.5 mm to 1.5 mm thickness

Annealing 1 hr at 600°C and pickling

Cold rolling at room temperature to 1 mm either in rolling direction or in the transverse direction

Annealing 1 hr at 600°C and pickling

Rolling at room temperature in the former direction to 0.5 mm

Annealing 1 hr at 600°C and pickling.

1.6.4 Fabrication of Pressure Tubes (ID 86/90 mm, 91/96 mm, 90/96 mm)

- Single extrusion from billets 90.5/255 mm and straightening for tubes of thickness higher than 2.5 mm
- Extrusion and drawing for the tubes with a thickness of 2 mm.

2. IDENTIFICATION OF THE FINISHED PRODUCTS

As was shown in Section 1.2, there is a direct correlation between the geometrical characteristics of the powder and the oxide content in wt.% but it can be seen (Ref. 69 and 84) that, with regard to the mechanical properties of the finished products, the oxidation rate is a less important factor than the geometrical characteristics, specially in the case of fine powders. For example, beyond a fixed oxidation rate (corresponding to an alumina film of 0.05 or 0.03 μ for particle thicknesses of 2 and 1 μ) an increase in the film thickness does not improve the properties of the sintered product.

Then, the finished products are not identified by their oxide content but in the same way as the powders, with, in some cases, the letter D as a final symbol to indicate that the product has undergone the vacuum degassing treatment (for example, A-120-20-D).

3. IMPROVEMENTS OF THE POWDERS

3.1 Intermediate Grades

The thicknesses of the normal powders are 12, 5 and 2 μ ; an attempt was made to employ some powders with thicknesses of 4, 6 and 9 μ but the mechanical properties of the finished products manufactured from these powders are similar to those of the main products.

3.2 Very Fine Powders

Some products were manufactured from powders of thickness 1 μ according to the following table.

Grade of powder	Alumina content of powder	Alumina content of finished product
A-10-01-D	3%	9.5%
A-10-03-D	9%	14%
A-10-05-D	13%	18%
A-10-07-D	22%	23%

The grade A-10-01-D, with the lower alumina content, gives products with a very fine dispersion which are very attractive because of either their mechanical resistance or their elongation properties (see figs. 16-20).

3.3 Improvement of the Starting Aluminium (Ref. 79, 80, 81, 82 and 85)

The products marked with the first symbol "A9" are manufactured from powder of high purity aluminium (99.9% Pure). With this pure aluminium the oxidation process is more difficult and it is necessary to increase the temperature in comparison with the corresponding process with the 99% pure aluminium.

The finished products exhibit the following properties:

- lower mechanical resistance than for the corresponding "A" product, and, correlatively, higher elongation to rupture (see Table 11);
- same creep properties (see Table 12);
- better aspect of the structure, very fine dispersion of alumina with little or no oxide clusters; this last property could be related to the more difficult oxidation process which gives a fine and regular oxide film.

3.4 Milling and Sizing of the Oxidized Powder (Ref. 79 and 81)

In order to reduce the number of oxide clusters in the structure, ball-milling of the oxided powder followed by pneumatic sizing is very effective:

- the milling eliminates the clusters of oxide formed during the oxidation process and maintains the typical lamellar structure of the powder;
- the sizing allows the last aggregates to be eliminated and gives an ultra-fine alumina powder (about 20% of the initial powder is eliminated by this sizing).

The products submitted to this special treatment are identified by the symbol BC (B for "broyage" = milling and C for "classification" = sizing).

Example: Frittoxal A - 10 - 01 - DBC

4. STRUCTURE - CHEMICAL COMPOSITION - DENSITY

4.1 Structure

The structure is typical of the composites Al-Al_2O_3: a fine dispersion of alumina in the aluminium matrix. Electron-microscopy shows the well-known cellular structure with subgrains of polygonal shape (fig. 6).

4.2 Chemical Composition

Some typical analyses are given in the following table:

Grade	Chemical composition (wt. %)				
	Al_2O_3	Fe	Si	Cu	Zn
A-60-20-D	9.5	0.39	0.14	0.01	0.01
A-60-30-D	13.0	0.38	0.14	0.01	0.01
A-90-15-D	5.1	0.42	0.13	0.01	0.01

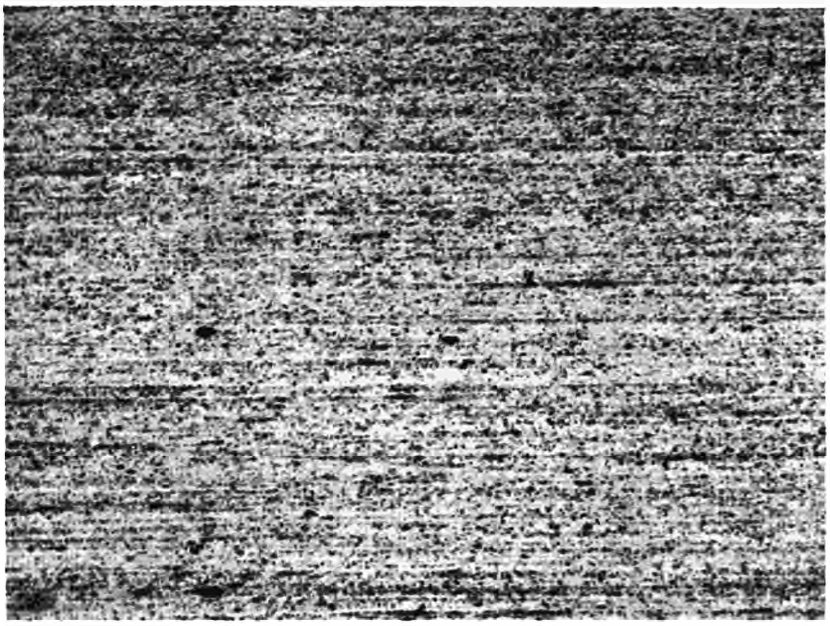

Fig. 5: Typical longitudinal microstructure of Frittoxal-grade A-50-10-D (enlargement x 450). Note the presence of clusters of alumina.

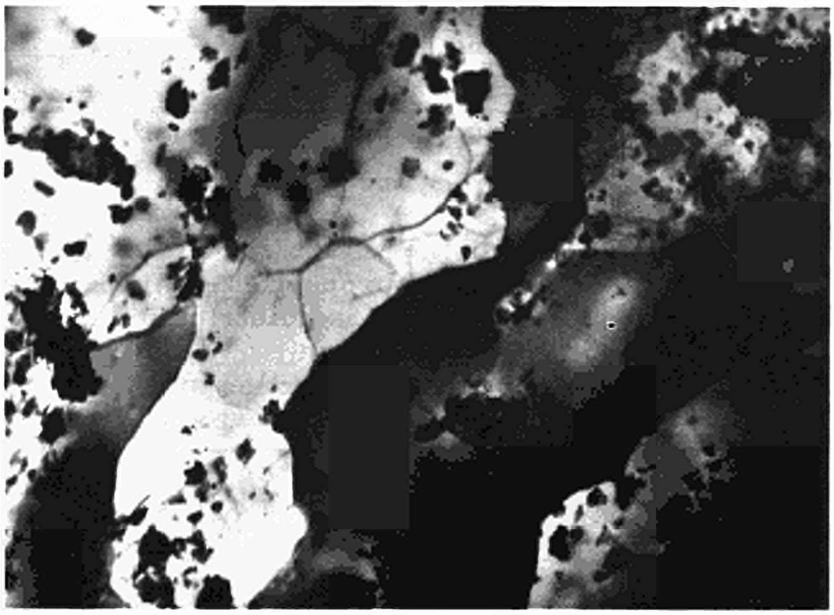

Fig. 6: Grade A-120-20-D
Electron-micrograph by transmission (enlargement x 7500) (Ref. 83)

4.3 Density of Finished Products (Ref. 41)

The density of the materials was determined by the method given in Ref. 39. The results are reported on the table below. The data are averages of three to six measurements.

Grade of Frittoxal	Density (g/cm^3)
A-120-20-B	2.73
A- 50-15-B	2.74
A- 20-07-D	2.77

5. MAIN PRODUCTS IN FRITTOXAL

In the following table details are given of the principal types of Frittoxal products studied at the TLH.

TABLE 1

Grade	Weight % Al_2O_3 average value		Purpose
A-120-20-D	5		
A-50-10-D	6		Cladding
A-20-07-D	10		Cladding) Pressure tubes) Main products
A-40-10-D	7		Mechanical properties similar to grade A-60-20
A-40-15-D	10		Mechanical properties similar to grade A-20-07
A-60-20-D	9.5		See A-40-10-D
A-60-30-D	13		Mechanical properties similar to grade A-50-10 (very coarse distribution of alumina)
A-90-10-D	3.6		) Mechanical properties similar to grade A-120-20
A-90-15-D	5.1		)
A-20-07-D	10		Study of the influence of the oxide
A-20-09-D	13		thickness on the mechanical properties
A-20-11-D	16		
	Powder	Finished product	
A-10-01-D	3	9.5	Study of the very fine powder
A-10-03-D	9	14	
A-10-05-D	15	18	
A-10-07-D	22	23	
A9-20-05-D	7		Study of the high purity aluminium as
A9-50-10-D	6		starting material

TABLE 2

Recall	Key to the identification of the product
1st symbol	starting aluminium A (99% purity) A9(99.9% purity)
1st figure	average thickness of the particles μ x 10
2nd figure	average thickness of the oxide film μ x 100
+D	vacuum degassing treatment
BC	eventually for ball-milling and sizing after the oxidation process
	Average Al_2O_3 content weight % $a = \frac{f}{e \times (0.00337)}$ f = thickness of the oxide film in μ e = thickness of the flakes in μ

6. MECHANICAL PROPERTIES OF FINISHED PRODUCTS OBTAINED FROM STANDARD GRADES OF FRITTOXAL

6.1 Tensile Tests

6.1.1 Test Specimens

In the TLH Laboratories (Ref. 72 and 73) several types of test specimens are used according to the shape of the product to be tested. Unless otherwise specified, their axes are taken along the direction of extrusion at the beginning or at the end of the extruded part.

Test specimens for rods

The dimensions of the calibrated part are as follows:

- diameter : 5 mm
- length : 80 mm

Test specimens for smooth tubes

The tube is first opened along its length, then flattened at 450°C by a rolling press, without reduction of thickness. Out of the sheet thus obtained, a piece is then machined as shown in fig. 7.

Test specimens for sheet material

The specimen is as thick as the sheet to be tested, its shape being shown in fig. 7.

Base length (Ref. 73)

In any case, the base length lo is equal to:

$$lo = \sqrt{67}\,S$$

S being the cross-section of the calibrated part. The length lo is measured between two engraved lines with a microscope (accurate to $\pm$ 0.01 mm).

Test specimens used in other laboratories

The test conditions adopted by the AEK Risö and the AECL laboratories have previously been described in Part I, Sections 3.2.3.1 and 3.2.2.1 respectively (see also Part I, fig. 2).

6.1.2 Testing Instruments (Ref. 73)

The TLH laboratories use a hydraulic testing machine (Amsler or Lhomme and Argy). The testing speed varies with the load but in no case does the test duration exceed 2 min, the standard mean speed being about 4 mm/min. For high temperature tests, the specimens are placed in a furnace regulated to within $\pm$ 3°C and the soaking time before testing is about 40 min.

6.1.3 Results of TLH Laboratories Tests

The results of tensile tests for the main grades of Frittoxal as a function of the temperature are given.

For each temperature the curve shows the highest and the lowest values, the point representing the main value. The engineering yield stress was always determined with the aid of the PP = f(ΔL) diagrams in continuous tests. Afterwards a straight line at 0.2% plastic elongation was drawn parallel to the elastic line of proportionality. It will be recalled that P represents the load and Δ L the cross-head displacement.

- Grade A-120-20-D (Ref. 73-84) fig. 8 and 9

  Tests have been conducted on extruded tubes annealed for 1 hr at 600°C. Every point on the curve is the arithmetical mean value of 22 measures for tests carried out at 20 and 500°C and of eight measures for tests at intermediate temperatures. The curves show that above 400°C Su mingles with So.2 and that the elongation (e_{pb}) reaches a minimum value between 450 and 550°C.

- Grade A-50-10-D (Ref. 73-84) fig. 10 and 11

  At 20 and 500°C, 28 measures have been carried out, and only five for each intermediate temperature. The results are similar as for the previous grade, but the strain seems to increase with temperature exceeding 500°C.

- Grade A-40-15-D (Ref. 77) fig. 12 and 13

  Only two samples have been tested for each temperature. This grade, which is very similar to the previous one gives similar results.

- Grade A-20-07-D (Ref. 81-85) fig. 14 and 15

  The number of tests performed for each temperature is not known.

- Grade A-10-01-D (Ref. 81-85) fig. 16 and 17

  The number of tests carried out is unknown.

  The results show that, as previously, the values of Su and So.2 mingle at high temperature and the elongation increases above 500°C. The zone between 500 and 600°C could be chosen for plastic transformation of the material (extrusion).

- Comparison of the five main grades of Frittoxal

  For each property all five grades of Frittoxal were compared (other presentation of previous results), fig. 18-20.

  The diagrams show clearly that for decreasing flake size, So.2 increases mainly at room temperature tests. Tests on two grades of close composition (A-50-10-B and A-40-15-D) yield similar values. The tensile stresses and the elongation decrease with increasing temperature; the minimum elongation always occurs at about 500°C and increases above.

6.1.4 Results of AECL Laboratories Tests

Tests have been carried out at AECL on products which have been named "Frittoxal 20-40-80" with oxide contents of 5.3, 4.25 and 1.25 wt.% respectively.

This old designation would approximately correspond to grades A-20-04, A-40-07 and A-80-15 respectively according to the formula in Table 2.

The results are reported in fig. 21, 21a, 21b, 22, 22a and 22b. The values of Su and So.2 above 400°C are in good agreement with the TLH ones, but the elongations are quite different, especially for the "40" and "80" grades (see fig. 18-20).

6.1.5 Results of Euratom Laboratories Tests

Some tensile tests at 450°C were performed on finned tubes of profile

ISML 3 (see Part I, Section 5. 2) extruded from billets of various grades (three test pieces for each grade). They were carried out on long pieces of tubes, the grips being out of the furnace, so the e_{pb} values are not available.

The values for Su are reported in Table 3.

6.1.6 Scatter of Results (Ref. 73)

Statistical studies concluded at TLH have shown that at 20 and 500°C there are no significant differences in the properties of materials taken at the beginning or at the end of extruded parts.

Consequently, the results obtained with all the samples can be considered as a whole.

Moreover, there is also no significant difference between different tubes.

The histograms in fig. 23 show the scatter of results given in diagramsä fig. 8-11 for the two grades A-120-20-D and A-50-10-D.

Note that the Su values are closer together than those for So. 2 and that the difference in strength for the two grades is greater at room temperature.

The mean scatters are reported in Table 4 (isolated values are eliminated).

6.2 Creep Tests

6.2.1 TLH Laboratories Tests (Ref. 73)

6.2.1.1 Test Conditions

The test specimen is the same as in fig. 7. However, for creep tests, the base length is measured with a microscope (accurate to ± 0.01 mm) between several microhardness prints. The elongations to be measured are so tiny that, even with this method, the error in the determination of strain can amount to 8-10%.

TABLE 3

Values of maximum tensile stress at 450°C on finned tubes

Grade of Frittoxal	% Al_2O_3	Su kg/mm^2	Extrusion ratio (1)
A-10-01-DBC	14.7	9.4	24
A-10-05-D	14.7	10.0	24
A9-10-01-DBC	10.0	10.5	24
A-20-07-DBC	13.4	6.8	1st extrusion: 3 2nd extrusion: 24
A9-20-07-DBC	16.9	8.5	24

(1) See Part I, Section 1.3.4.

TABLE 4

Scatter in tensile properties (in %)

Grade of Frittoxal	Test Temperature					
	20°C			500°C		
	Su	So.2	e_{pb}	Su	So.2	e_{pb}
A-120-20-D	13.5	36	43	32	43	62
A-50-10-D	10	16	53	31	40	67

6.2.1.2 TLH Laboratories Results

All the results available in the progress reports have been summarized and are given for each grade of Frittoxal.

Grade A-120-20-D

The curves in fig. 24 show the variation in elongation after 100 hr as a function of the applied stress (Ref. 73-84). Note that the elongation remains very small up to an applied stress C1 which decreases rapidly for increasing test temperature. The elongations to rupture are always low (about 0.5%).

It seems more interesting to sum up these results as mean values in Table 5 below.

TABLE 5

Stresses causing a given elongation after 100 hr at various temperatures (Frittoxal A-120-20-D) Ref. 84

Test temp. (°C)	Stress giving elongation 0.2% (kg/mm^2)	Stress giving an elongation of between 0.2 and 0.5% (kg/mm^2)
450	0.8	1.4
500	0.6	1.0
550	0.1	0.5

At 450°C a stress of 1.3 kg/mm^2 produces 0.35% elongation in 1000 hr (Ref. 78). Fig. 25 contains a histogram for the elongation after 100 hr at 500°C for 14 extruded tubes from two different batches of powder. A statistical study has shown that there is no significant difference between two groups of results and that the standard deviation does not exceed 0.1%. The stresses causing rupture after 100 hr at 450, 500 and 550°C are 1.7, 1.2 and 0.8 kg/mm^2 respectively (Ref. 73-84).

Grade A-50-10-D

The few results we have gathered are reported on Table 7 below.

TABLE 7

Stresses (in kg/mm^2) giving in H hours an elongation A(%) on Frittoxal A-40-10-D

Elongation A(%)	Time H (hours)	Test Temperature kg/mm^2 450°C	500°C	Ref.
0.1	100	1.4		77
	1,000	1.3	1.1	
0.2	100		1.2	
0.25	100	1.75		78
0.35	1,000	1.6		

Grade A-20-07-D

Fig. 26 (Ref. 75) contains a stress-to-rupture curve plotted with only three results to give a rough idea of the behaviour of the material. At 450°C the following values for stress and elongation can be considered for engineering evaluation purposes (Ref. 85).

Stress leading to rupture in 100 hr	: 2.8 ± 0.2 kg/mm^2
Elongation at rupture in 100 hr	: 1.5 ± 0.5%
Stress causing 0.5% elongation in 1000 hr	: 2.1 ± 0.2 kg/mm^2

Grade A-10-01-D

The only values available at 450°C are listed below (Ref. 85)

Stress leading to rupture in 100 hr	: 4.5 ± 0.2 kg/mm^2
Elongation at rupture in 100 hr.	: 1.5 ± 0.5 kg/mm^2
Stress causing 0.5% elongation in 1000 hr	: 3.5 ± 0.2 kg/mm^2

Comparison of the five grades of Frittoxal

Table 8 gives the creep properties of the five grades.

TABLE 8

Creep properties at 450°C of the five grades of Frittoxal

Grade of Frittoxal	Stress producing rupture in 100 hr (kg/mm^2)	Elongation at rupture in 100 hr (%)	Stress giving an elongation of between 0.2 and 0.5% in 1000 hr	Stress giving 0.5% elongation in 1000 hr (kg/mm^2)	Ref.
A-120-20-D	1.7	0.5	1.3		84
A-50-10-D	1.85	0.5	1.5		84
A-40-10-D			1.6		78
A-20-07-D	2.8 ± 0.2	1.5 ± 0.5		2.1 ± 0.2	85
A-10-01-D	4.5 ± 0.2	1.5 ± 0.5		3.5	85

Note once again that fine grain products are more interesting for both strength and elongation.

6.2.2 AECL Laboratories Tests (Ref. 33)

Fig. 27 shows a stress-to-rupture curve obtained at AECL on 'Frittoxal 40" (probably a grade A-40-06; See Section 6.1.4).

6.3 Burst Tests on Finned Tubes (Euratom Results)

Some tubes have been tested to rupture in a furnace at 450°C under internal gas pressure. As for the ISML products, the pressure increase was about 10 kg/cm.^{2}/min and only results obtained under these conditions were taken into consideration.

Table 9 gives tangential stress values calculated from the following:

$$\sigma_t = \frac{p \cdot D}{2e}$$

σ_t = tangential stress (kg/mm^2)
p = pressure at rupture (kg/mm^2)
D = internal diameter of the tube (mm)
e = minimal wall thickness (mm)

For comparison we have also reported the values for the longitudinal rupture stress already given in Table 3.

TABLE 9

Tangential and longitudinal rupture stresses on finned tubes at 450°C

Grade of Frittoxal	Tangential stress (σ_t) kg/mm^2	No. of test pieces	Longitudinal stress (kg/mm^2)	Extrusion Ratio(1)
A-10-01-DBC	7.6 ± 0.3	2	9.4	24
A-10-05-D	6.9 ± 0.2	2	10.0	24
A9-10-01-DBC	7.1 ± 0	2	10.5	24
A-20-07-DBC	7.7 ± 0.7	3	6.8	1°extr.:3 2° " :24
A9-20-07-DBC	8.5 ± 0.6	3	8.5	24

(1) See Part I, Section 1.3.4.

6.4 Hardness Tests

Some Brinell hardness tests were carried out at TLH (Ref. 73) on A-120-20-B grade under the following conditions:

load	: 5 kg
Ball diameter	: 1 mm

Tests carried out on 30 specimens gave a value of 40 ± 3. Microhardness tests carried out at AECL (Ref. 86) using a 33 g load yielded the following results:

Frittoxal 20	: 74
Frittoxal 40	: 58
Frittoxal 80	: 43

6.5 Young's Modulus (Ref. 73)

This has been determined on the classical Le Rolland-Sorin pendular elasticimeter on two specimens taken from different rods. The mean value obtained was:

$$6900 \pm 20 \text{ kg/mm}^2$$

7. INFLUENCE OF STARTING PRODUCTS

In order to improve the finished products, the characteristics of the starting products were varied in order to study the influence of the flake thickness, the oxide thickness and the purity of the starting aluminium.

7.1 Influence of Flake Thickness (Ref. 85)

Only the mean thickness of the flakes is taken into consideration. The tensile properties at 20, 400 and 450°C as a function of flake thickness obtained in the TLH and AEK Risö laboratories are given in fig. 28 and 29 respectively. The oxide thickness ranged between 0.07 and 0.10 μ. The curve in fig. 30 shows the stresses causing rupture after 100 hr at 450°C for various flake thicknesses. The strength of the material decreases with increasing flake thickness in both short-time tests (tensile) and long-time tests (creep).

The same results appear clearly also on cumulative diagrams of tensile tests (fig. 18-20), the more interesting grades being those with the finest particles (A-10-01-D and A-20-07-D).

7.2 Influence of Oxide Thickness (Ref. 85)

Fig. 31 gives the results for tensile tests at 20 and 450°C as a function of the oxide thickness for four flake thicknesses. It can be seen that the oxide thickness has less influence than the flake thickness. However, for the finest powders (1 μ), an increase in the oxide thickness above 0.02 μ reduces the strength. The creep tests (fig. 32) lead to the same conclusion.

7.3 Purity of Starting Aluminium (Ref.80-85)

It seemed to be worthwhile reducing the percentage of impurities in the starting aluminium in order to increase the corrosion resistance, to reduce the scatter of the mechanical properties and to lower the neutron absorption. In order to verify these points, experimental extruded products were prepared from aluminium of purity A 9 (99,9% purity) instead of A 4 (99.0% purity). Typical analyses of the main impurities in finished products obtained from A 4 and A 9 aluminium are given in Table 10.

TABLE 10

Main Impurities in finished products obtained from A 4 and A 9 aluminium

Grade of Frittoxal	Type of starting aluminium	Fe %	Si %	Al_2O_3 %	Ref.
A-20-07-D	A 4	0.44	0.30	12.4	41 (1)
A-50-15-B		0.47	0.11	7.8	
A-120-20-B		0.45	0.08	5.4	
A9-20-05-D	A 9	0.070	0.047	8.84	70-85
A9-50-10-D		0.062	0.048	5.36	

(1) The experimental procedure for chemical analysis is explained in detail in Ref. 41.

The results of tensile tests at 20 and 450°C are reported in Table 11.

TABLE 11

Tensile properties of materials obtained from Aluminium A 4 and A 9

Grade of Frittoxal	Type of starting aluminium	20°C tests			450°C			Ref.
		Su kg/mm^2	So.2 kg/mm^2	e_{pb} %	Su kg/mm^2	So.2 kg/mm^2	e_{pb} %	
A-20-07-D	A 4	23	16	8	4.8	4	6	85
A-50-10-B		19.5	15	6	4	3.5	4	84
A9-20-05-D	A 9	19.2	12	13.2	4.2	4	6.2	79
A9-50-10-D		15.7	10.4	17	3.6	3.3	11	

The strength of products prepared from purer aluminium are 10 to 20% lower than those obtained from current aluminium powder.

The results of the creep tests are reported on Table 12.

Note that the creep properties are practically identical for the two types of aluminium.

TABLE 12

Stresses and elongations at rupture after 100 hr at 450°C for materials obtained from Aluminium A 4 and A 9

Grade of Frittoxal	Type of starting aluminium	Stress at rupture (kg/mm^2)	Elongation at rupture (%)	Ref.
A-20-07-D	A 4	2.8 ± 0.2	1.5 ± 0.5	85
A-50-10-D		1.85		73
A9-20-05-D	A 9	2.75	1	80
A9-50-10-D		2.1	1.6	80

8. INFLUENCE OF HEAT TREATMENTS

All specimens previously considered were annealed for 1 hr at 600°C. It was interesting to study the influence of such a treatment and to analyse separately the effects of the annealing temperature and time.

8.1 Influence of the Annealing Temperature (Ref. 74-85)

Fig. 33 shows the evaluation of the tensile properties with increasing temperatures and Table 13 the reduction in % of strength and elongation. As in fig. 13, note the following points for tests carried out at 25°C.

- the material with coarser grains is more affected by the treatment;
- the values of So.2 are reduced further than those for Su;
- the higher the annealing temperature, the lower the mechanical properties.

The properties at 500°C are affected in a more complicated way.

8.2 Influence of the Annealing Time (Ref. 76-85)

Specimens of two grades of Frittoxal were annealed at 450°C for 1, 10, 100 and 1000 hr. Fig. 34 shows the curve for the mechanical properties versus the annealing time. The stresses tend to drop very slightly, but are constant for fine-grade Frittoxal. The elongation shows no significant variation.

8.3 Effect of a Long-Time Annealing at High Temperature (Ref. 77)

Annealing treatments at 500 and 600°C were carried out up to 1000 hr. Significant differences were only noticed for treatments at 600°C. The variation in the mechanical properties versus annealing time is reported in the curves in fig. 35, and the reduction of strength in Table 14.

TABLE 14

Reduction of mechanical properties after an annealing treatment of 1000 hr at 600°C

Grade of Frittoxal	Tensile tests at 20°C		Tensile tests at 450°C	
	$\frac{Su - S'u}{Su}$ (%)	$\frac{So.2 - S'o.2}{So}$ (%)	$\frac{Su - S'u}{Su}$ (%)	$\frac{So.2 - S'o.2}{So.2}$ (%)
A-120-20-D	11	25	12	12
A-50-10-D	2	2	8	10

9. INFLUENCE OF MECHANICAL TREATMENTS

The cold deformation ratio H' for sheet material is defined by the manufacturer as:

$$H' = \frac{T - t}{t} \times 100$$

H' : ratio of cold-deformation in % for sheet material

T : starting thickness

t : final thickness

The cold-deformation ratio H for drawn tubes has been previously defined (Part I, Section 1.4).

Fig. 36 gives the mechanical properties of rods, tubes and sheets of Frittoxal A-50-10-D versus the deformation ratio. It is also shown that an annealing treatment of 1 hr at 600°C removes the cold hardening (the same values are obtained as in the curves in fig. 10 and 11). Note that the mechanical properties decrease above a deformation ratio of 500%.

10. INFLUENCE OF THE ANISOTROPY (Ref. 74, 75 and 85)

The mechanical properties previously reported were determined on samples taken along the direction of extrusion. However, the properties relating to the transverse direction are very important for cladding tubes undergoing an internal pressure.

TABLE 13

Reduction of mechanical properties after annealing at 450 and 600°C

Grade of Frittoxal	Test temp. (°C)	Material annealed 1 hr at 450°C			Material annealed 1 hr at 600°C		
		$\frac{Su-S'u}{Su}$ [1] %	$\frac{So.2-S'o.2}{So.2}$ %	$\frac{e_{pb}-e'_{pb}}{e_{pb}}$ %	$\frac{Su-S'u}{Su}$ %	$\frac{So.2-S'o.2}{So.2}$ %	$\frac{e_{pb}-e'_{pb}}{e_{pb}}$ %
A-120-20-B	25	3.8	9.2	-23.5	8.3	33	- 46
	500	3.3	0	9	9.8	0	13.5
A-50-10-B	25	1	4.3	-8.8	1	12	- 20.6
	500	5	2.8	-21	10	20	- 9

(1) Symbols with apostrophe refer to annealed specimens.

Microspecimens were cut out of smooth tubes (diameter 10/11.6 mm) longitudinally and transversally, with various base lengths cause difficulty in the interpretation of the results.
The following conclusion can be drawn:

For each property, we can define a ratio R (%) of diminution as for the maximum tensile stress (example below):

$$R = \frac{S_l - S_t}{S_l} \times 100$$

S_l = maximum longitudinal tensile stress
S_t = maximum transverse tensile stress

The values of R for tests carried out at room temperature are given in Table 15.

TABLE 15

Reduction of mechanical properties in the transverse direction (at 20°C)

Grade of Frittoxal	Values of R	
	for Su (%)	for e_{pb} (%)
A-120-20-D	12	30
A-50-10-D	6	
A-20-07-D	1 to 2	

The anisotropy drops with decreasing flake thickness. In high temperature tests, the anisotropy vanishes above 400°C.

11. Thermal Cycling (Ref. 75-85)

Thermal cycling tests were carried out between 300 and 500°C on specimens of grade A-120-20-D and A-50-10-D.
The test conditions were as follows:

- heating up to 300°C in a nitrate bath oven at 300°C;
- fast transfer to an oven at 500°C (time of 0.5 to 1 sec);
- stand-by of the specimen for 10 sec;
- return to the oven at 300°C.

The temperatures of oven were regulated to ± 5°C. After 2000 cycles, no significant changes either in the mechanical properties at 20 and 500°C or in the creep properties were noticed.

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CAPTIONS OF FIGURES

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3	Cross-section of oxidized particles (grade A-50-10)	
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13	Tensile tests on Frittoxal A-40-15-D	$e_{pb} = f(T)$
14	Tensile tests on Frittoxal A-20-07-D	$S = f(T)$
15	Tensile tests on Frittoxal A-20-07-D	$e_{pb} = f(T)$
16	Tensile tests on Frittoxal A-10-01-D	$S = f(T)$
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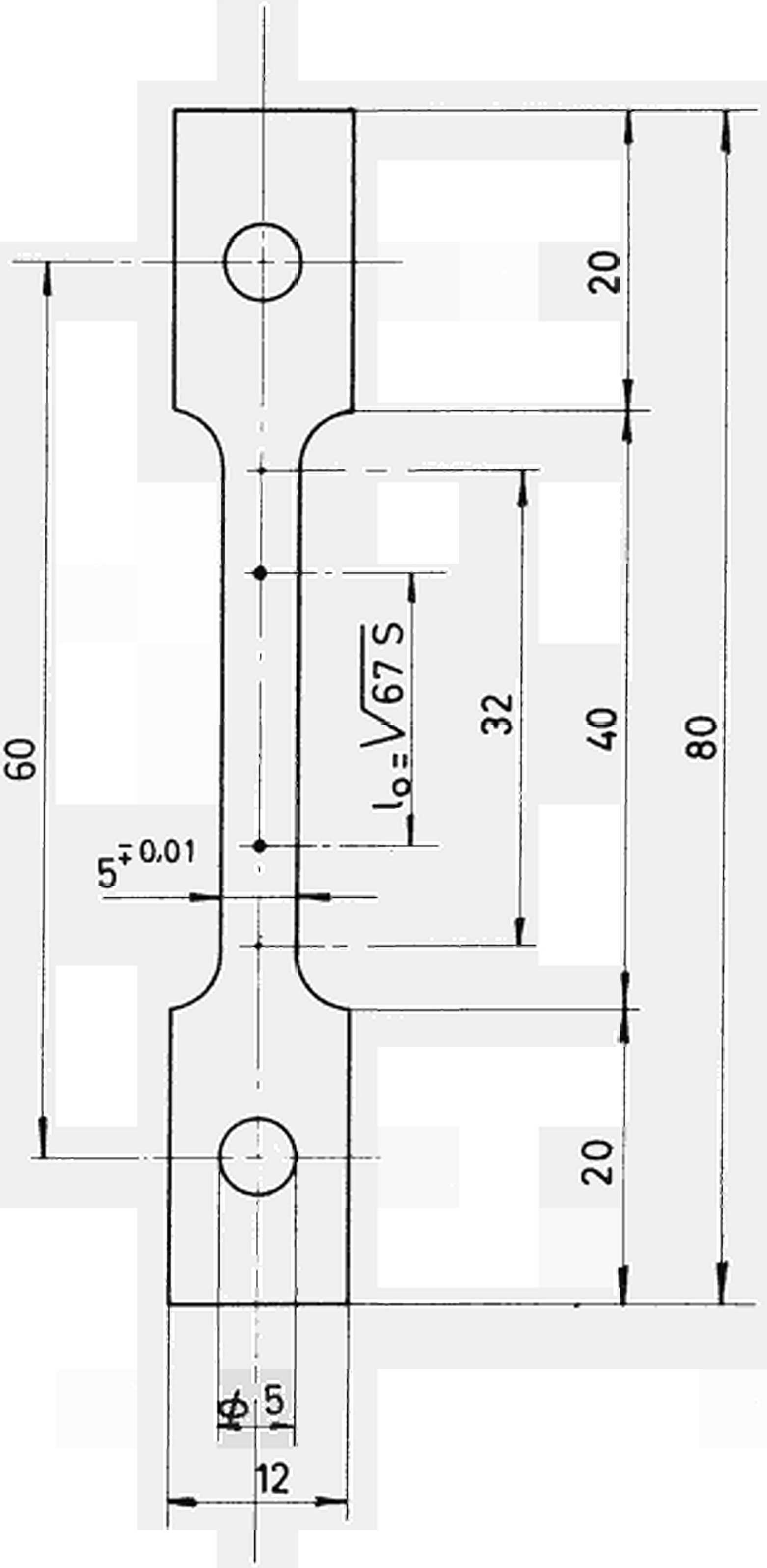
CAPTIONS OF TABLES

Table

Fig. 7

T. L.H. Tensile test specimen for tubes ands sheet materials

(Ref. 73)



Tensile tests on FRITTOXAL A 120-20-D

(Ref. 73-84)

Stress S=f(T)

Material: tubes 10/11,6 annealed 1H/600°C

Batch N^{os} 295-315

Specimen Fig. 7

⊡ $S_{0,2}$ (Engineering 0.2 offset yield stress)

⊙ Su (Engineering maximum tensile stress

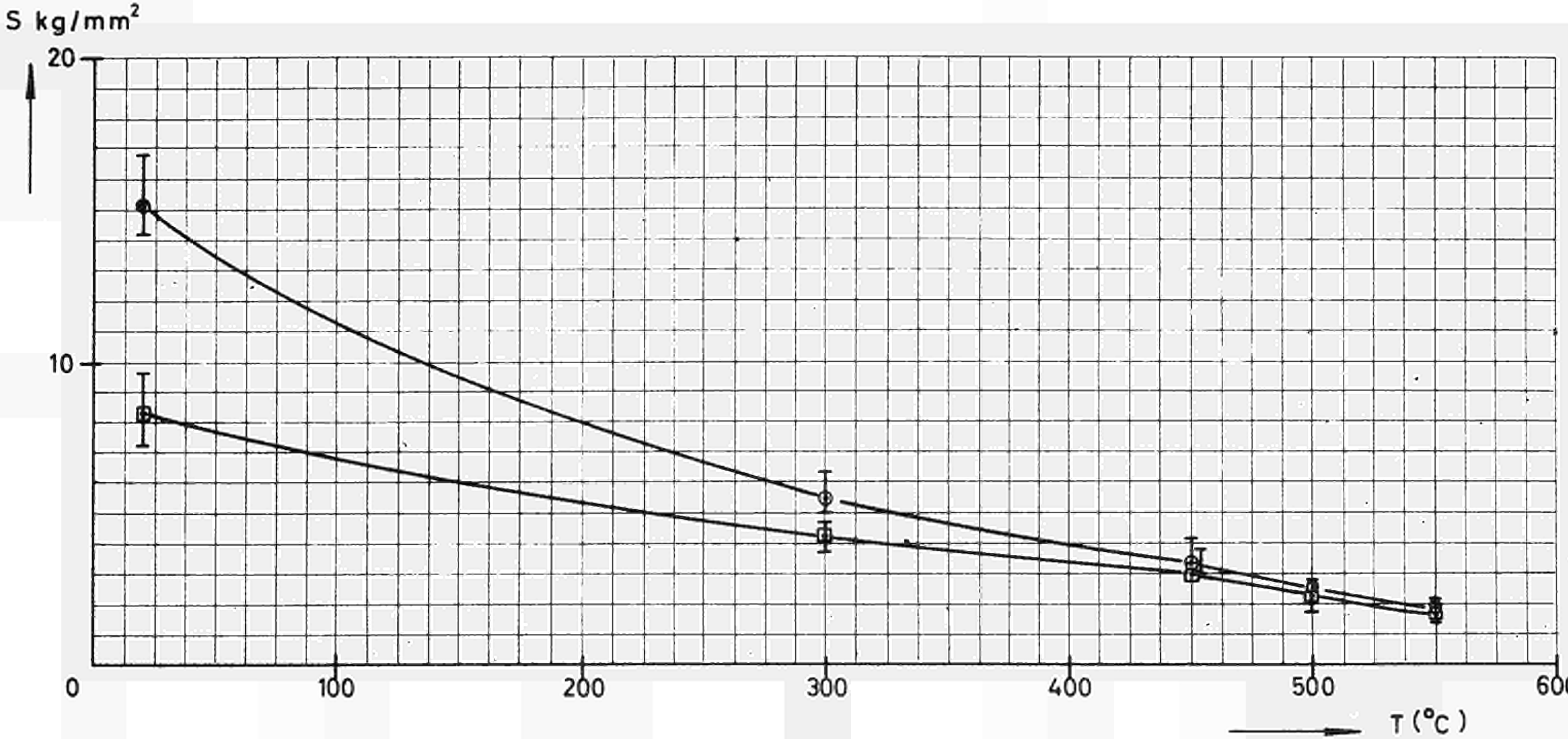


Fig. 8

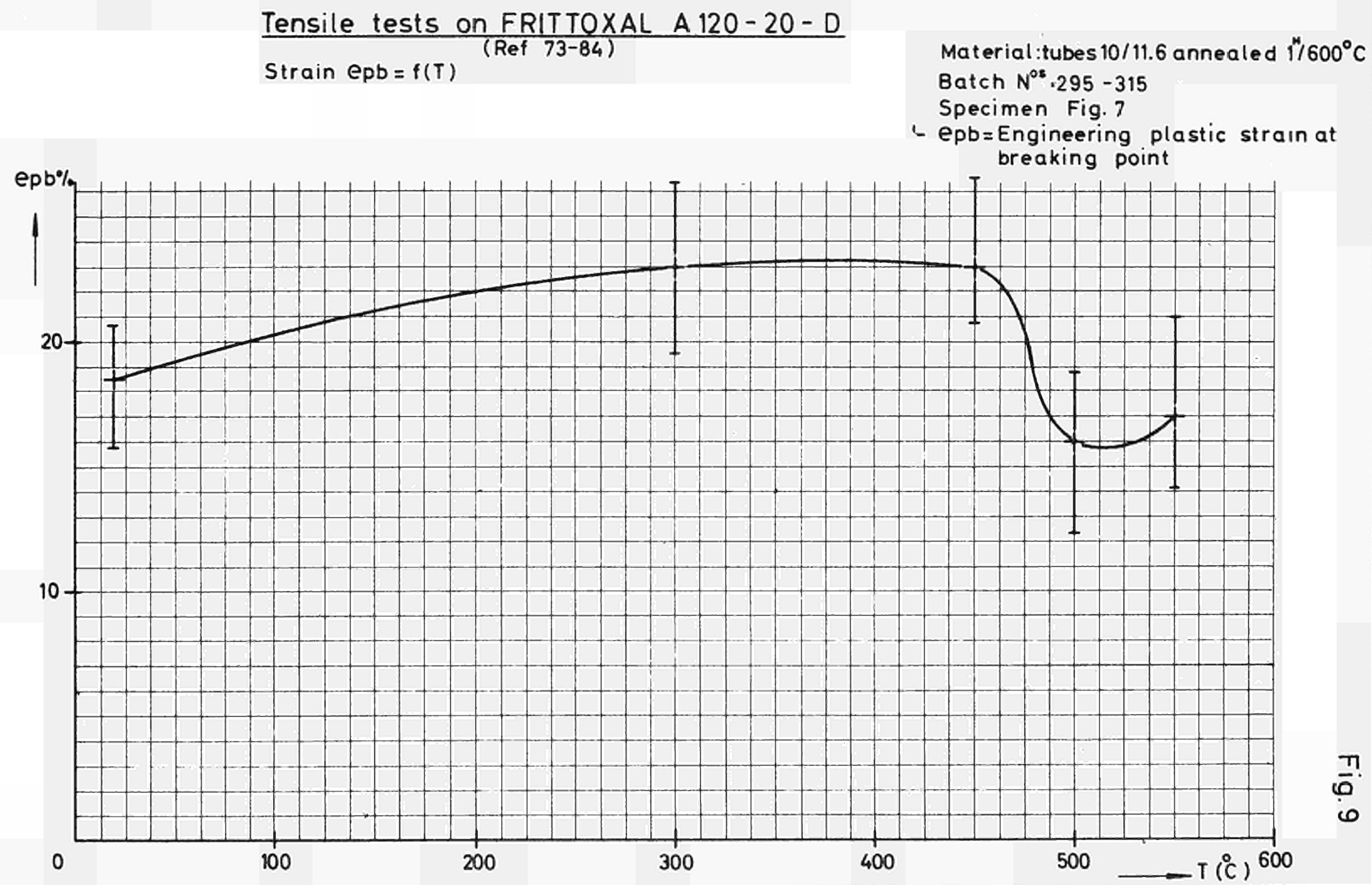

Tensile tests on FRITTOXAL A 120 - 20 - D
(Ref 73-84)
Strain epb = f(T)
Material: tubes 10/11.6 annealed 1 h/600°C
Batch N^{os} 295 - 315
Specimen Fig. 7
epb = Engineering plastic strain at breaking point
epb%
20
10
0
100
200
300
400
500
600
T (°C)

Fig. 9

Tensile tests on FRITTOXAL A-50-10-D

(Ref. 73-84)

Stress S = f (T)

Material : tubes 10 / 11.6 annealed 1^H / 600°C

Batch N^os 327

Specimen Fig. 7

⊙ Su (Engineering maximum tensile stress)

⊡ $S_{0,2}$ (Engineering 0.2 offset yield stress)

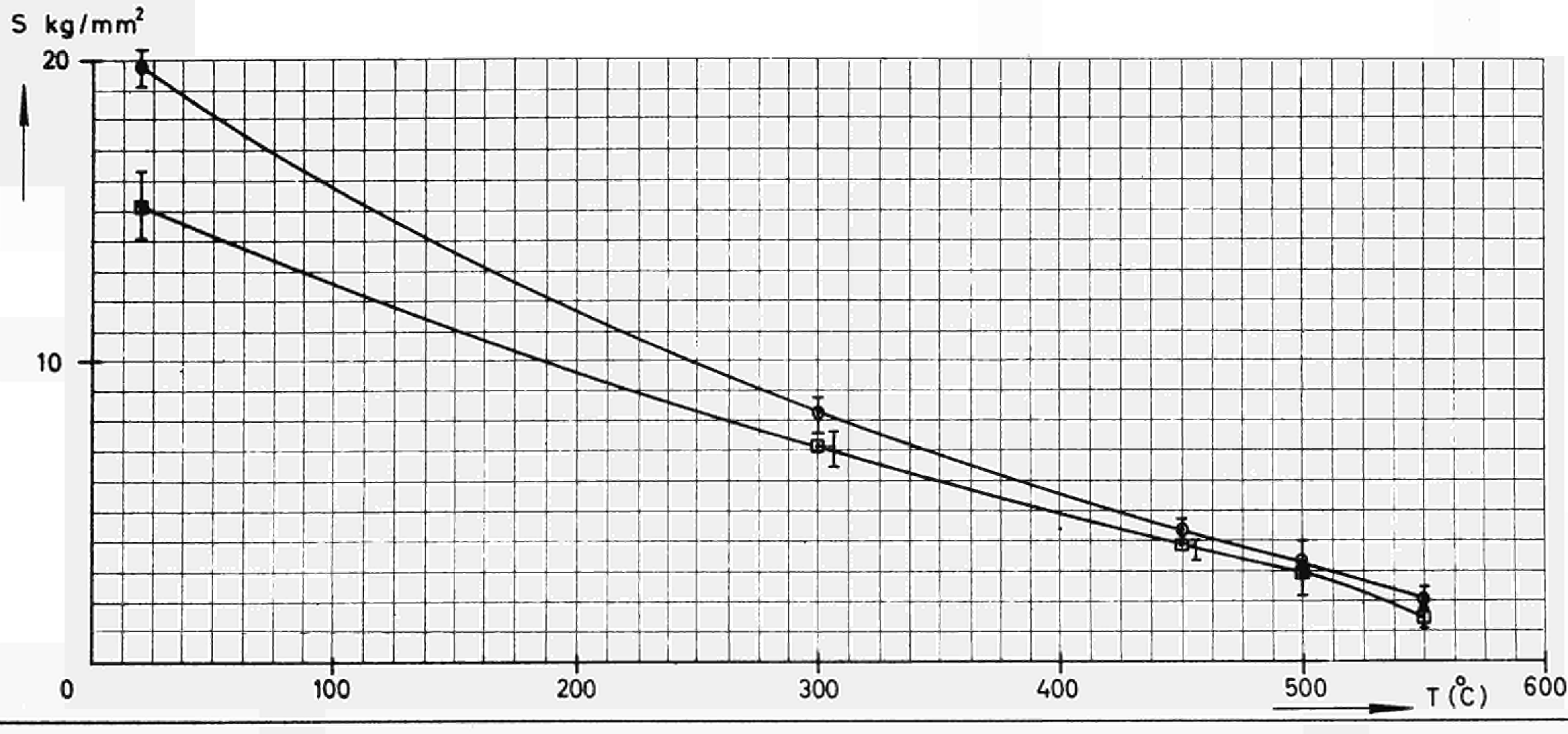


Fig. 10

Tensile tests on FRITTOXAL A-50-10-D
(Ref. 73-84)

Strain epb = f(T)

Material: tubes 10 / 11.6 annealed 1^H / 600° C

Batch N^os 327

Specimen Fig. 7

+ epb (Engineering plastic strain at breaking point)

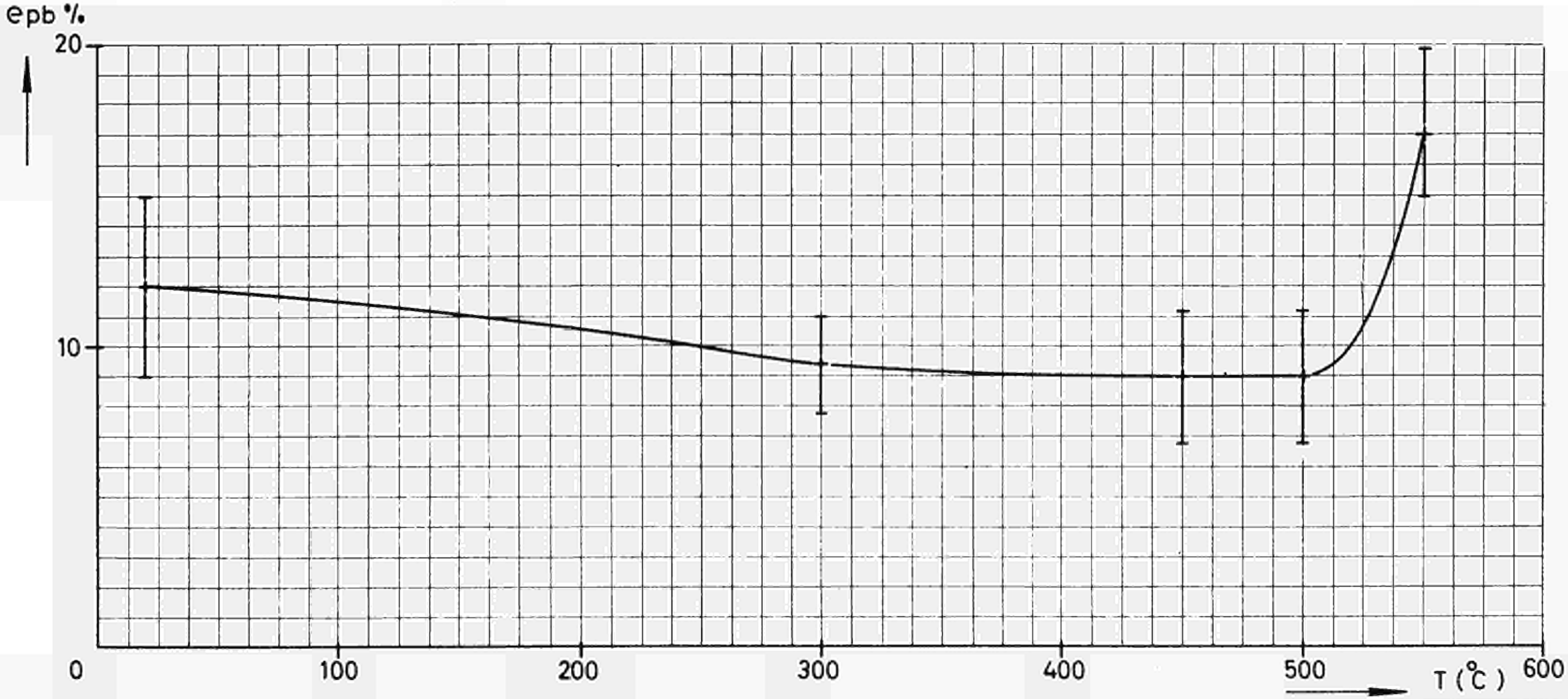


Fig. 11

Tensile tests on FRITTOXAL A-40-15-D
(Ref. 77)

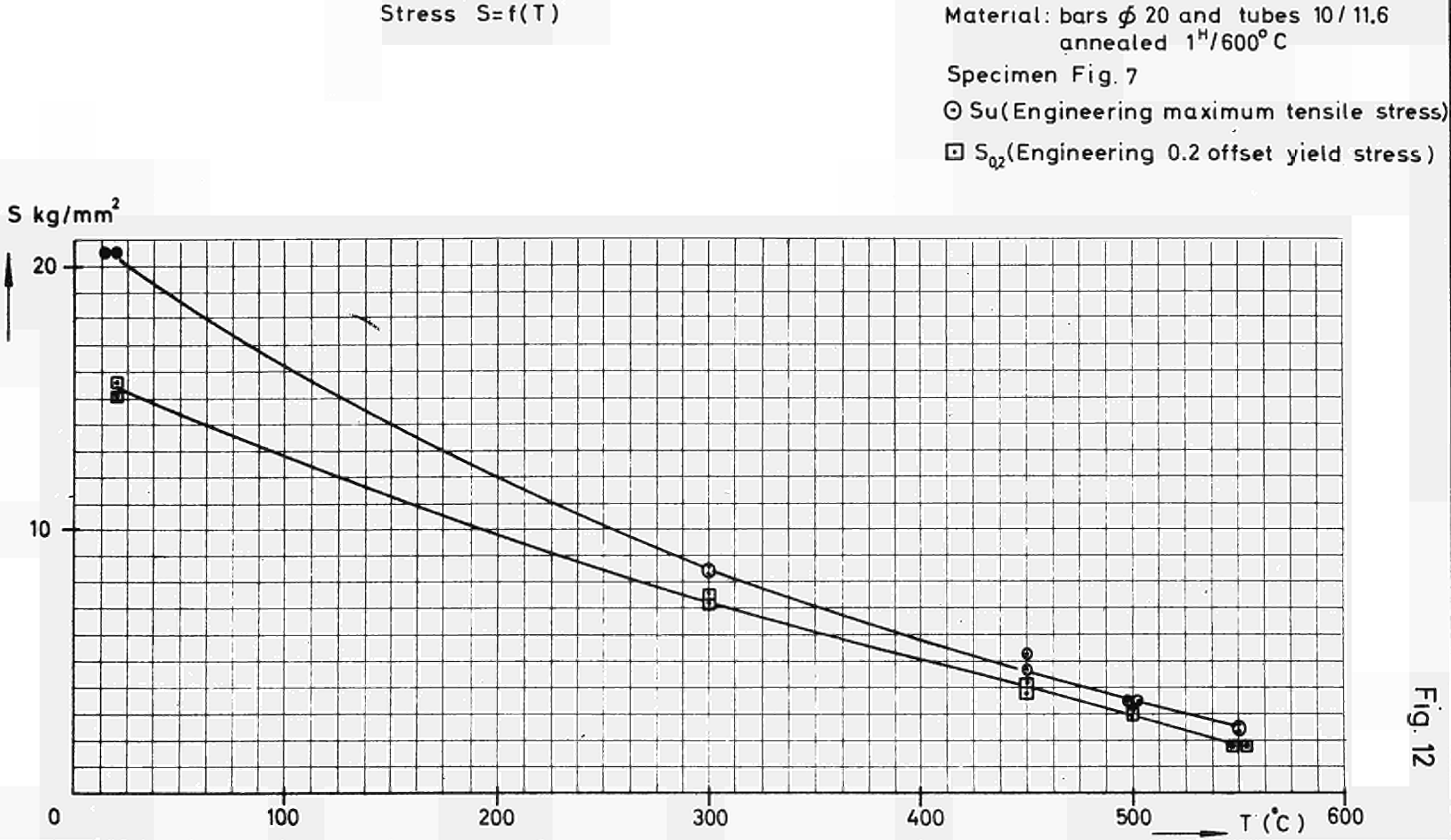


Fig. 12

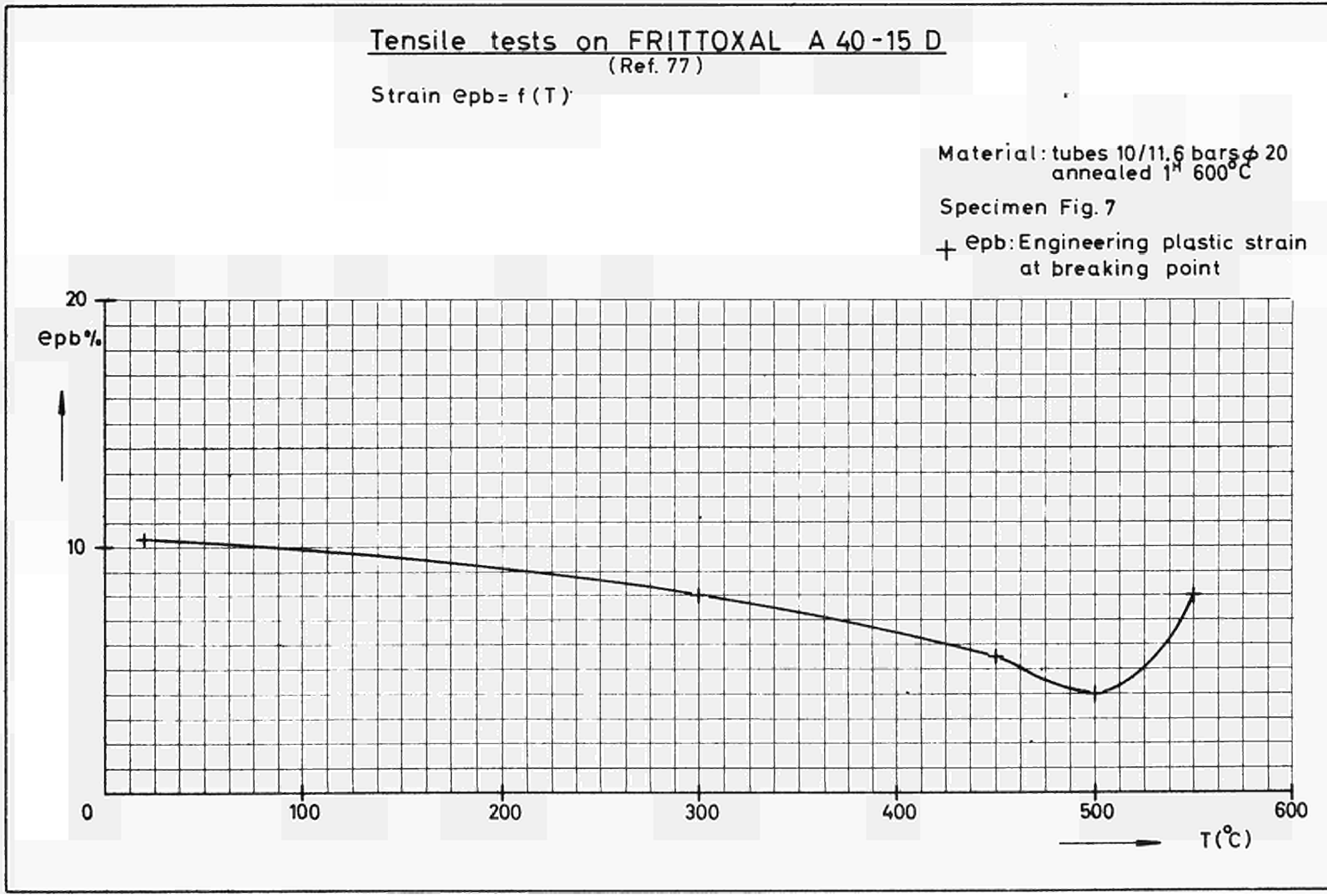
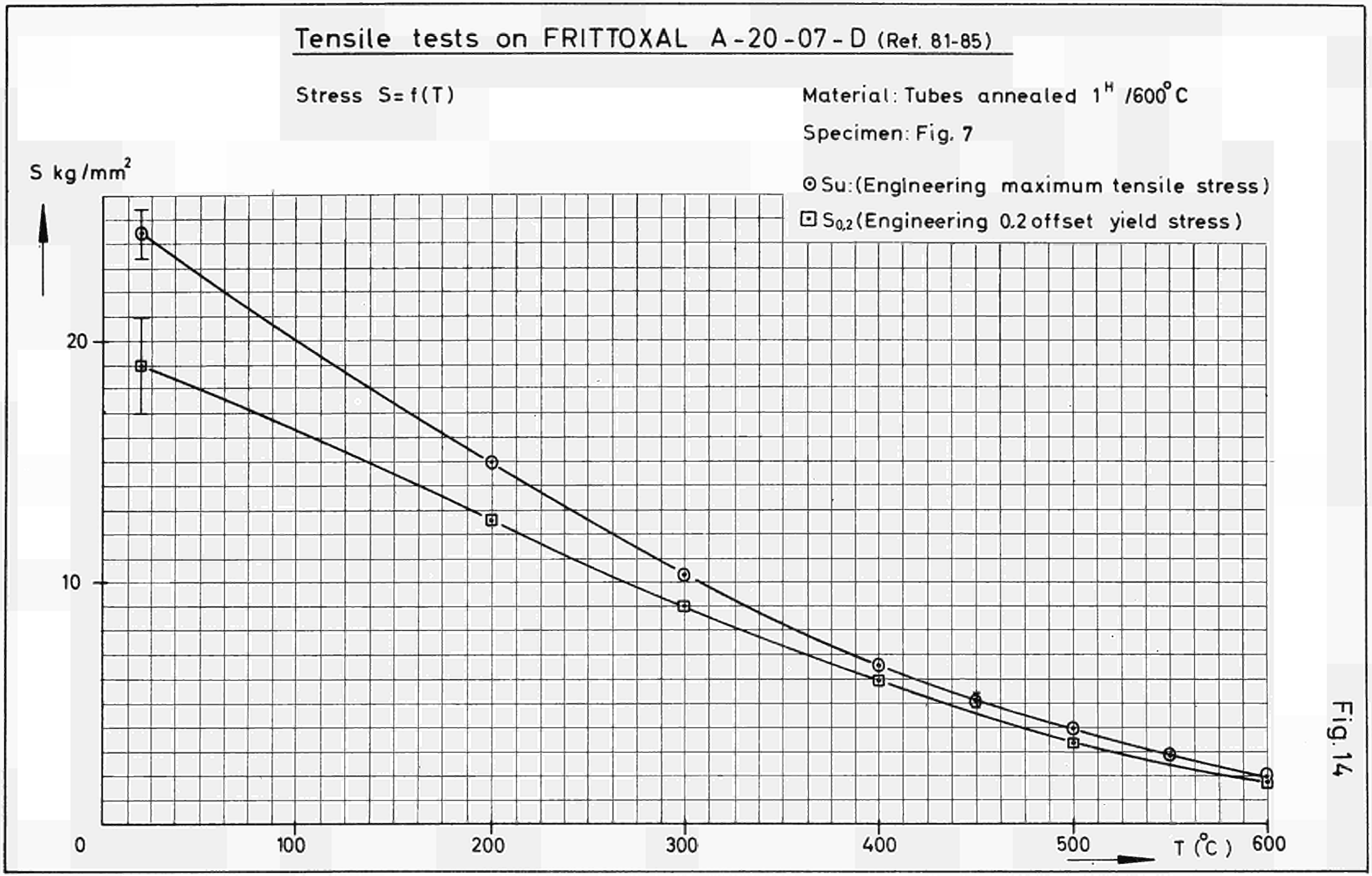

Tensile tests on FRITTOXAL A 40-15 D
(Ref. 77)
Strain epb= f (T)
Material: tubes 10/11.6 bars ϕ 20
annealed 1h 600°C
Specimen Fig. 7
+ epb: Engineering plastic strain
at breaking point
20
epb%
10
0
100
200
300
400
500
600
T(°C)

Fig. 13



Tensile tests on FRITTOXAL A-20-07-D (Ref. 81-85)
Stress S=f(T)
Material: Tubes annealed 1H /600°C
Specimen: Fig. 7
⊙ Su: (Engineering maximum tensile stress)
⊡ S0,2 (Engineering 0.2 offset yield stress)
S kg/mm2
20
10
0
100
200
300
400
500
600
T (°C)

Fig. 14

Tensile tests on FRITTOXAL A-20-07-D

Ref. (81-85)

Strain epb = f(T)

Material: tubes annealed $1^H/600°C$

Specimen Fig: 7

+ epb = (Engineering plastic strain at breaking point)

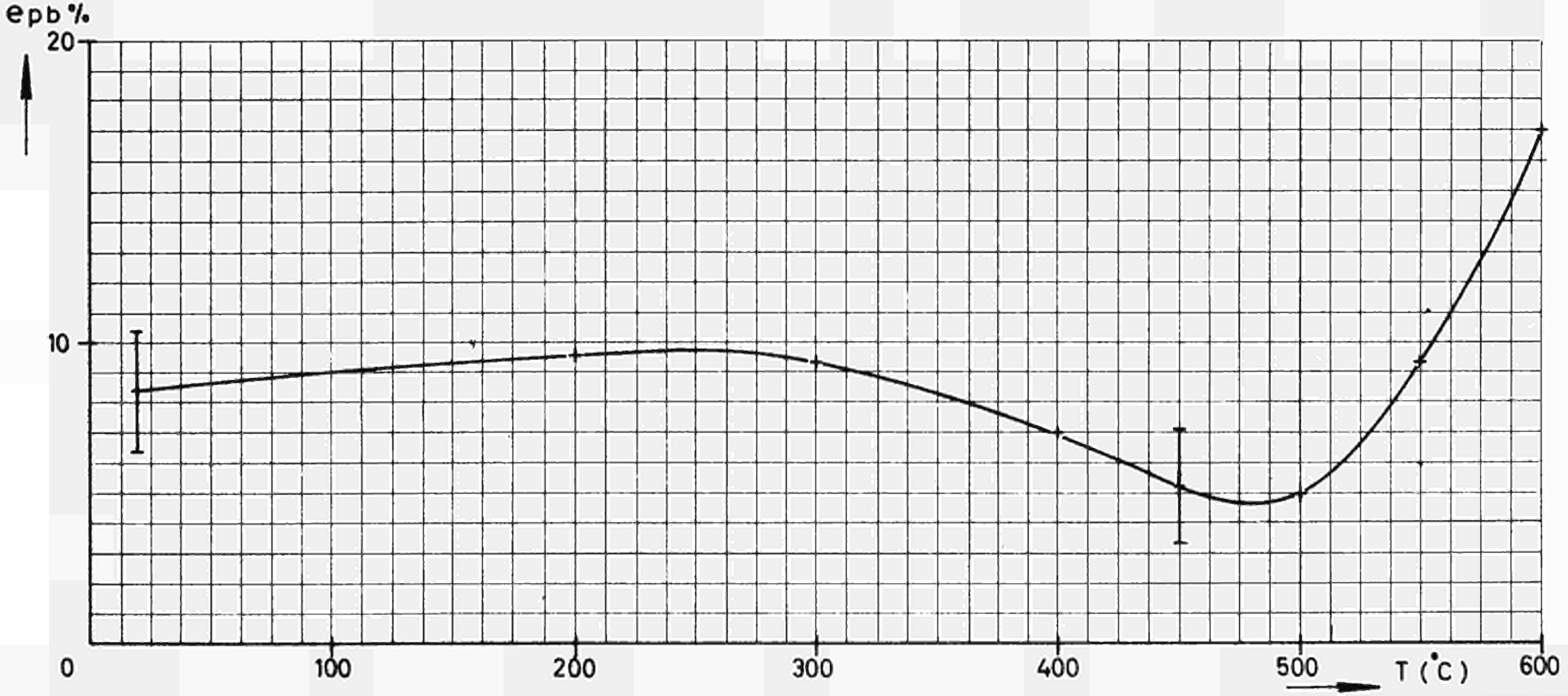


Fig. 15

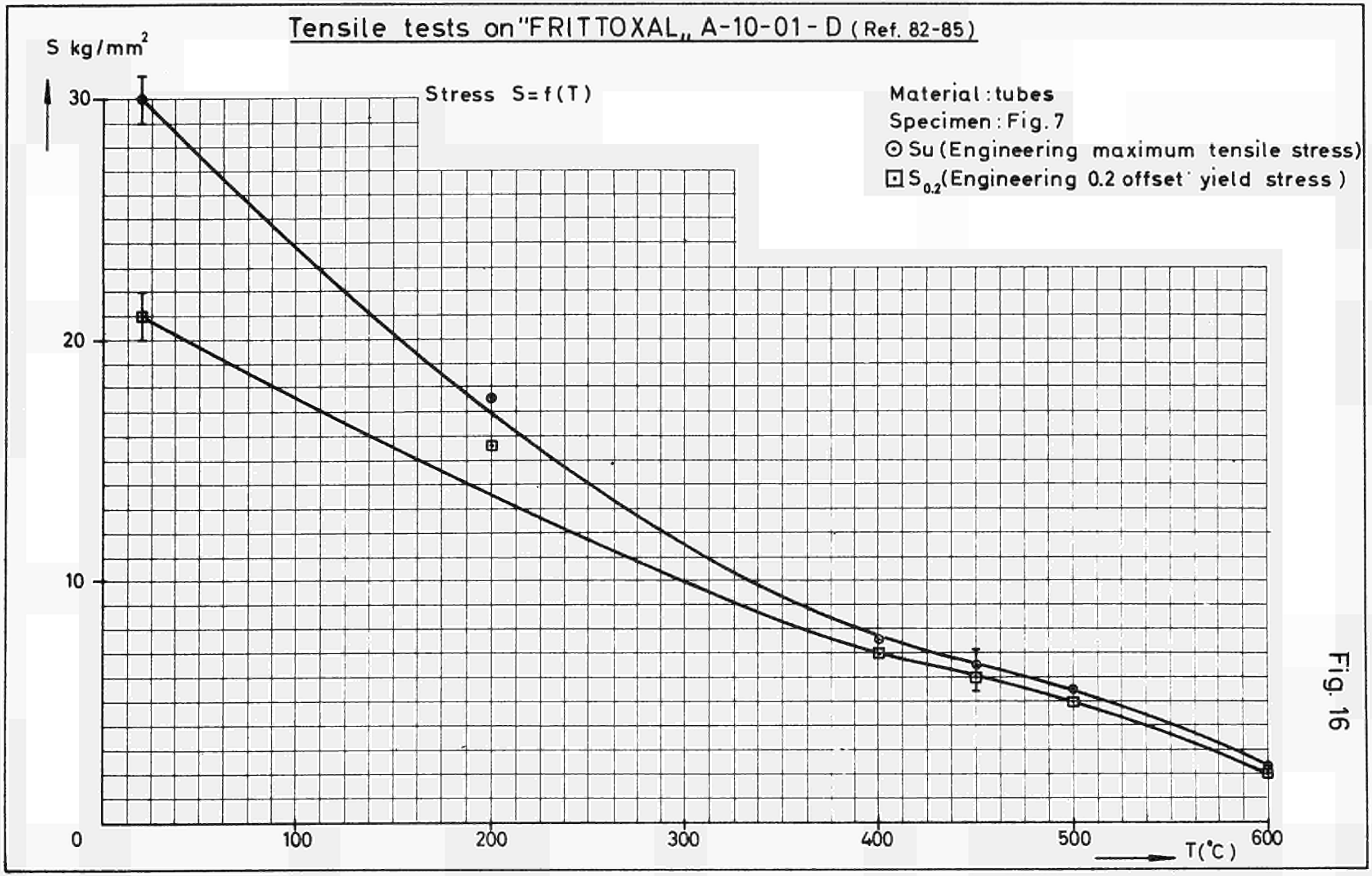

Tensile tests on "FRITTOXAL" A-10-01-D (Ref. 82-85)
S kg/mm²
Stress S=f(T)
Material: tubes
Specimen: Fig. 7
Su (Engineering maximum tensile stress)
S0.2 (Engineering 0.2 offset yield stress)
30
20
10
0
100
200
300
400
500
600
T(°C)

Fig. 16

Tensile tests on "FRITTOXAL" A-10-01-D (Ref. 82-85)

Strain epb = f (T)

Material : tubes
Specimen : Fig 7
+ epb = (Engineering plastic strain at breaking point)

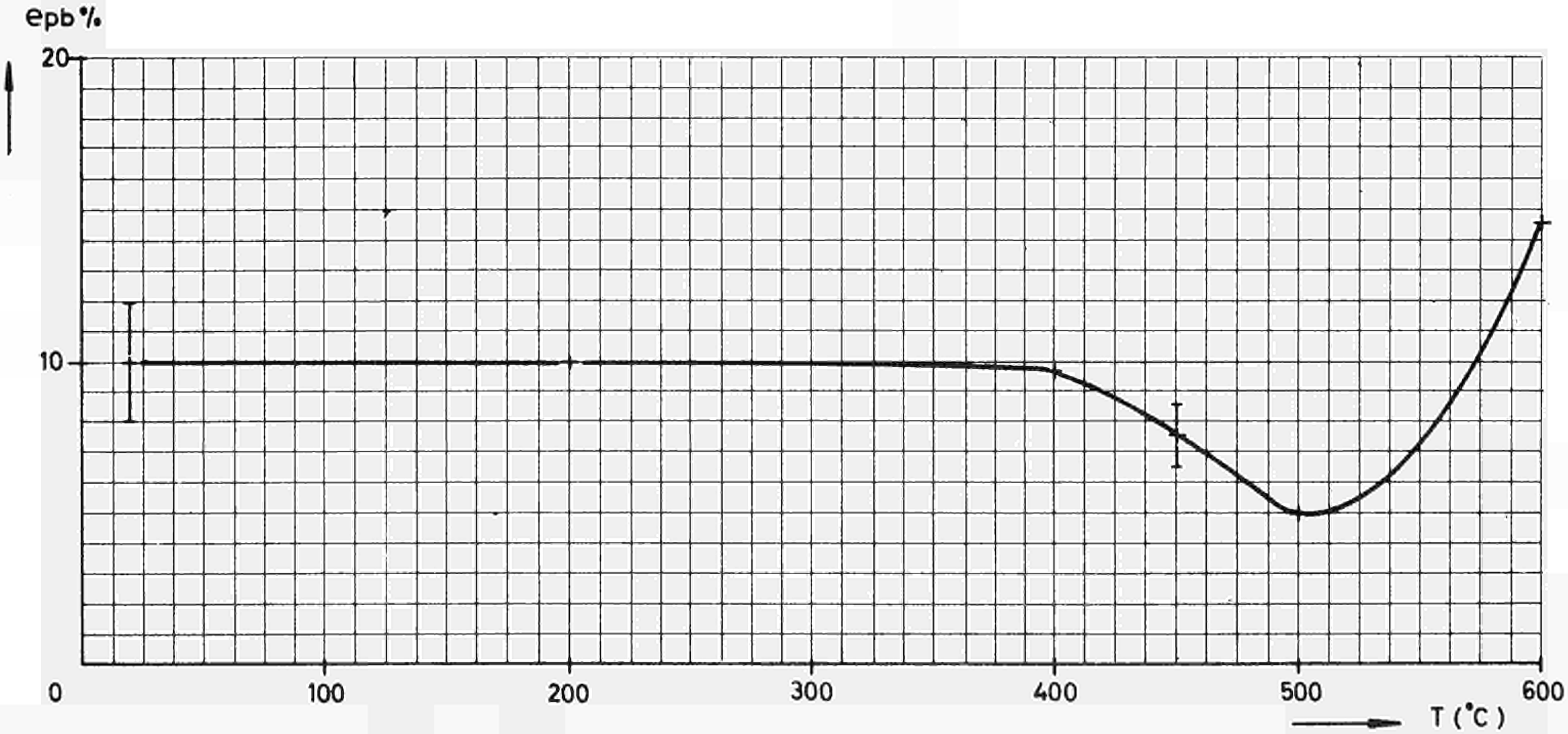


Fig. 17

Tensile tests on FRITTOXAL

Cumulative diagrams

Su= f(T)

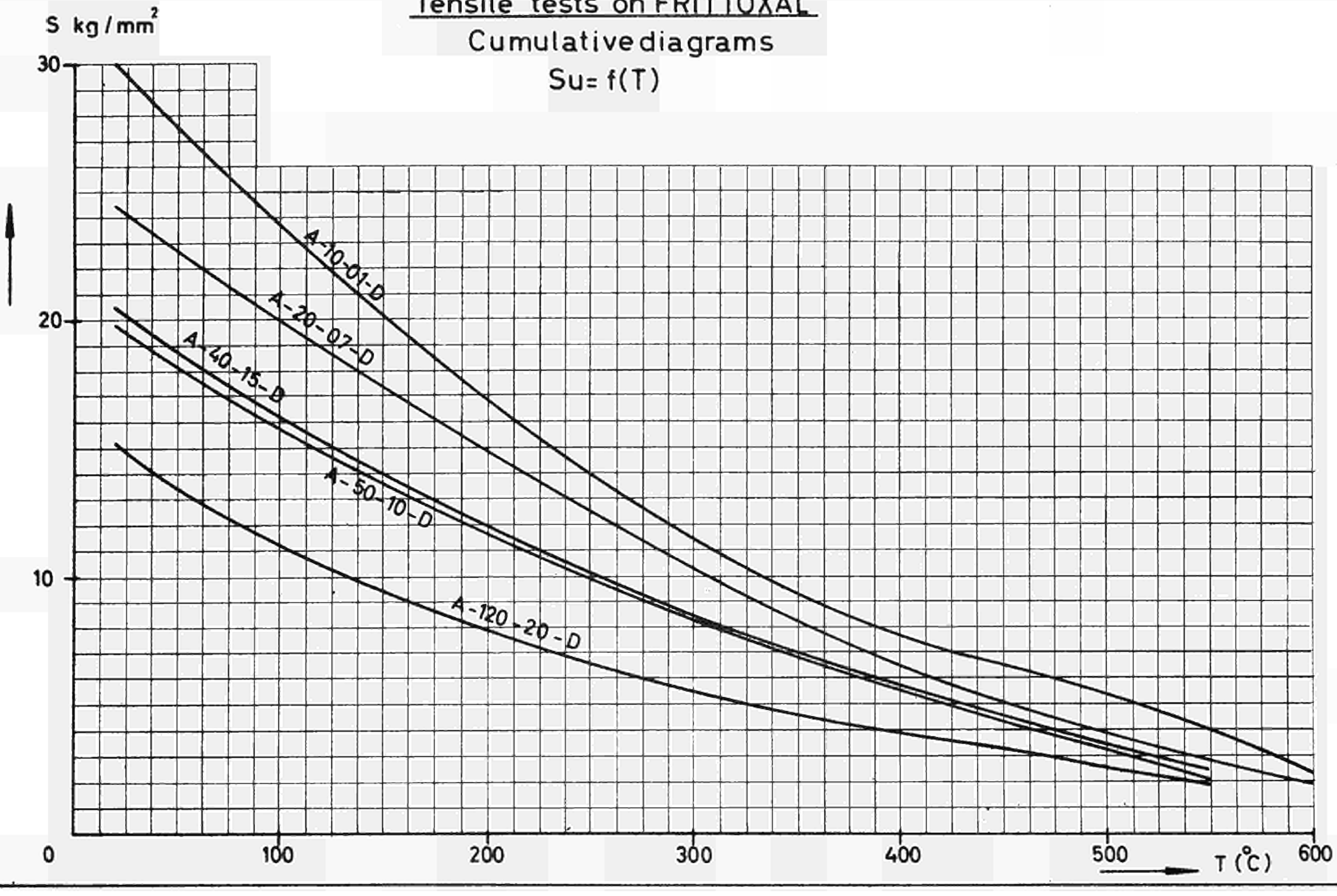


Fig. 18

Tensile tests on FRITTOXAL

Cumulative diagrams

$S_{0,2} = f(T)$

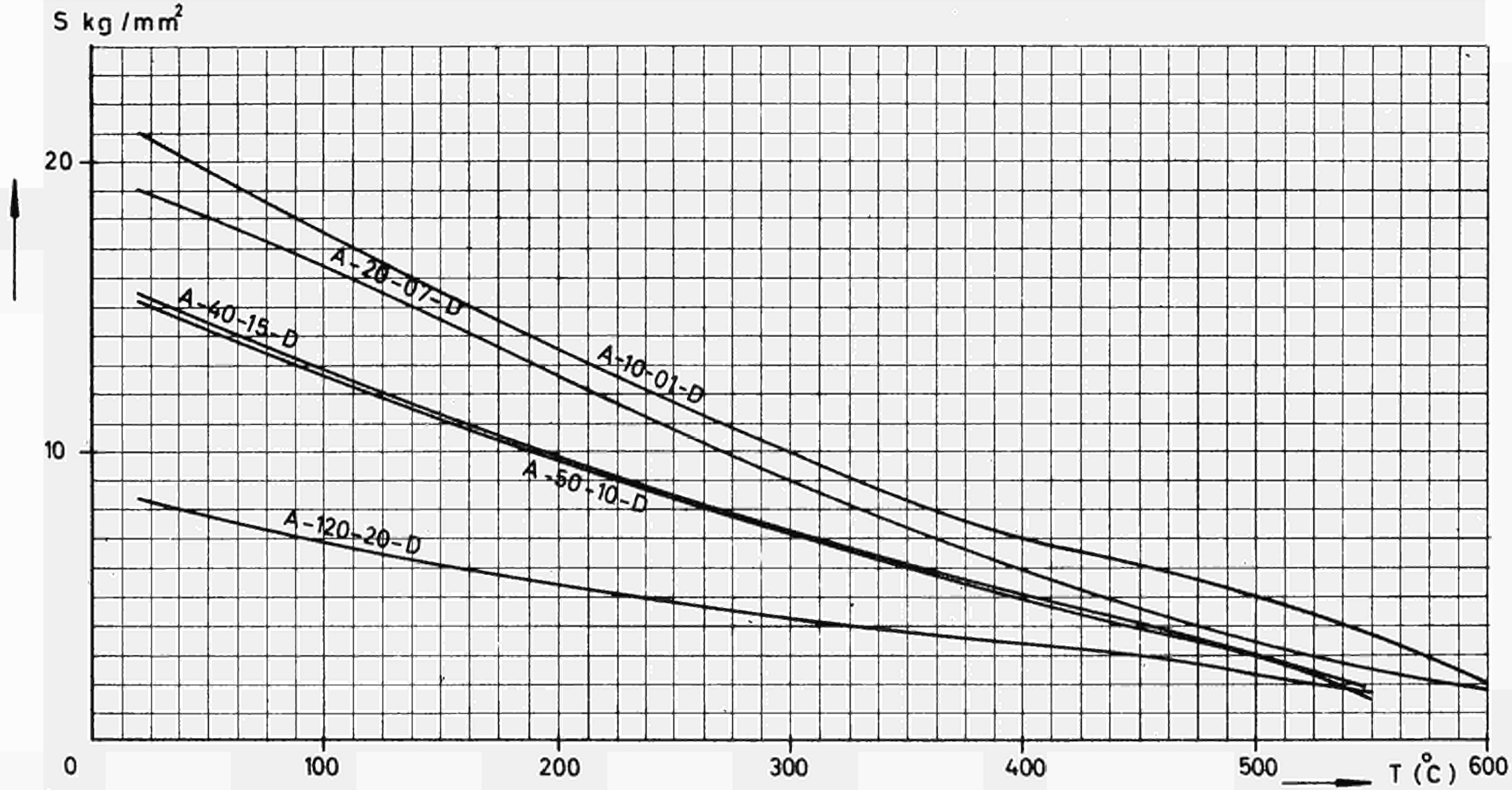


Fig. 19

Tensile tests on FRITTOXAL
Cumulative diagrams
$e_{pb} = f(T)$

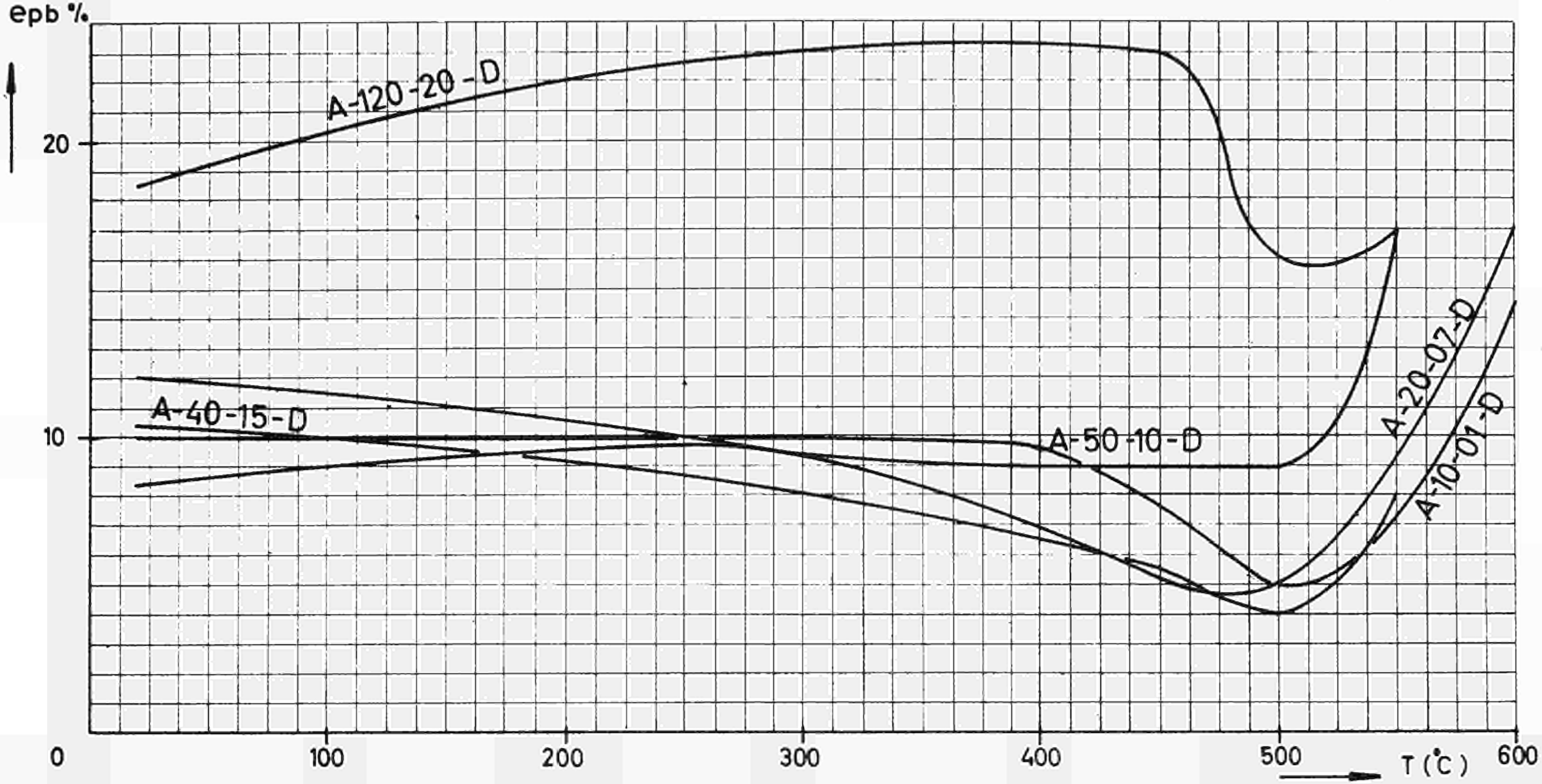


Fig. 20

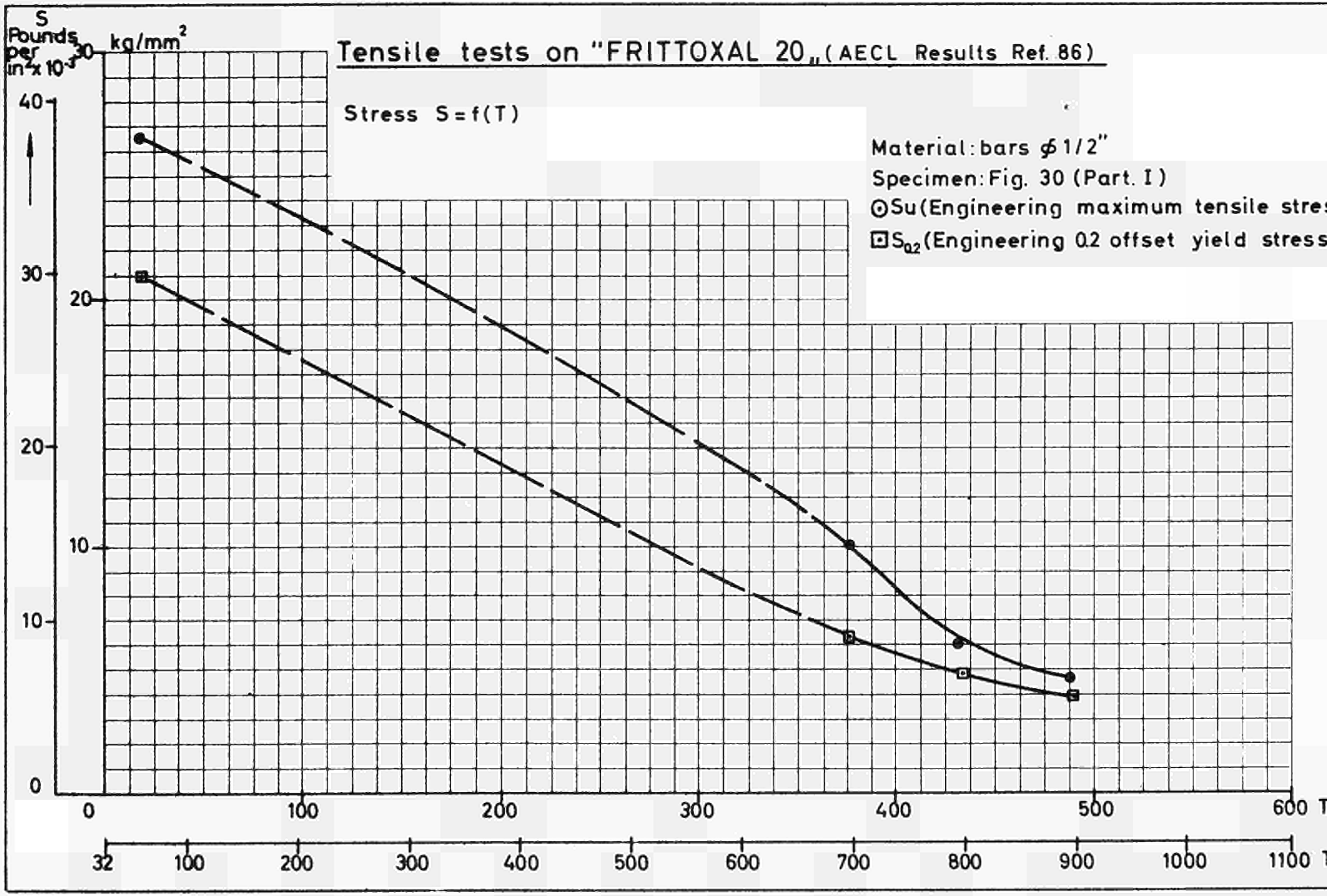

Tensile tests on "FRITTOXAL 20" (AECL Results Ref. 86)
Stress S = f(T)
Material: bars ∅ 1/2"
Specimen: Fig. 30 (Part. I)
⊙ Su (Engineering maximum tensile stress)
⊡ $S_{0.2}$ (Engineering 0.2 offset yield stress)
S Pounds per in² x 10⁻³
kg/mm²
0
10
20
30
40
T(°C)
0
100
200
300
400
500
600
T(°F)
32
100
200
300
400
500
600
700
800
900
1000
1100

Fig. 21

Tensile tests on „FRITTOXAL 40" (AECL. Result Ref. 33-86)

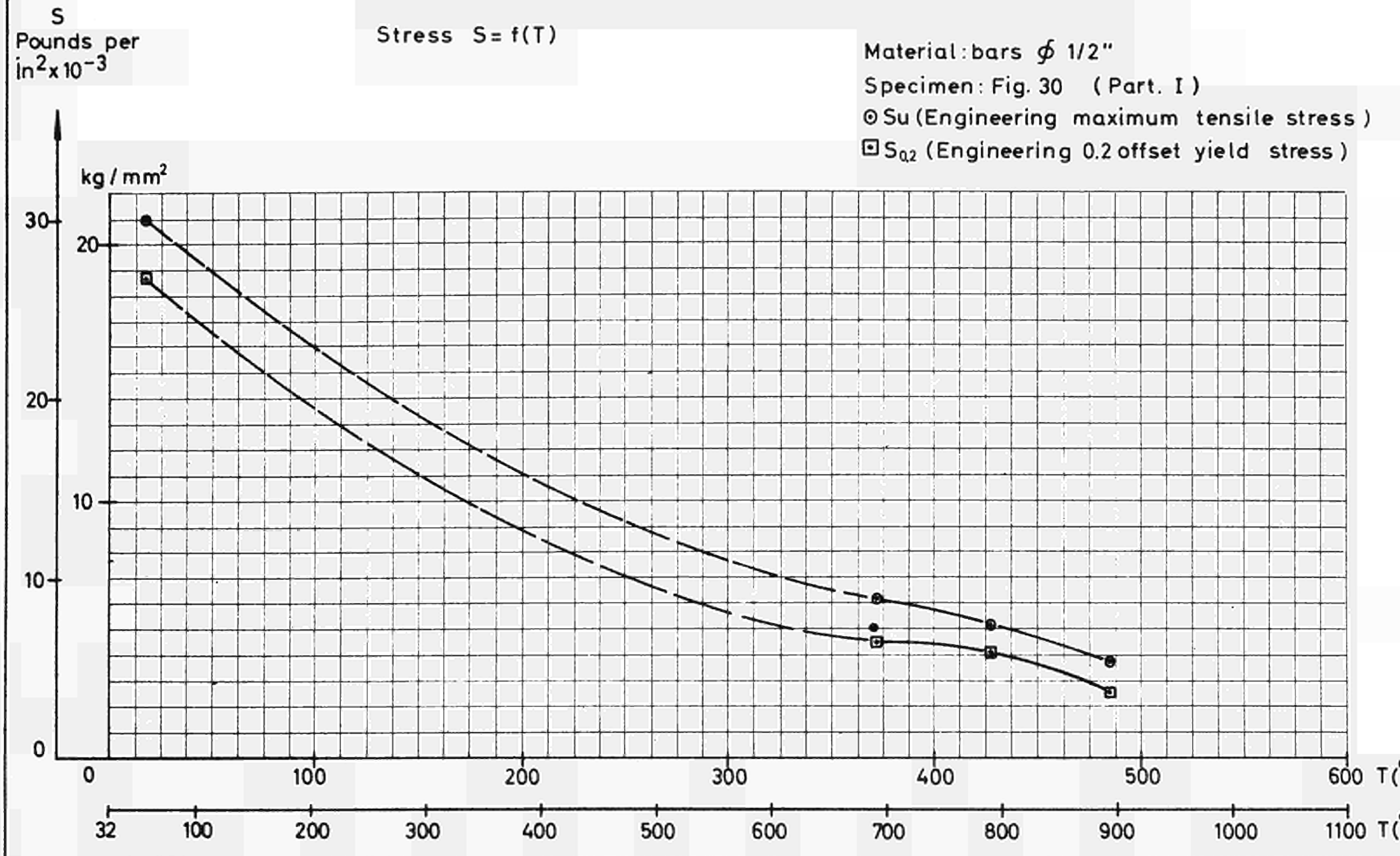


Fig. 21a

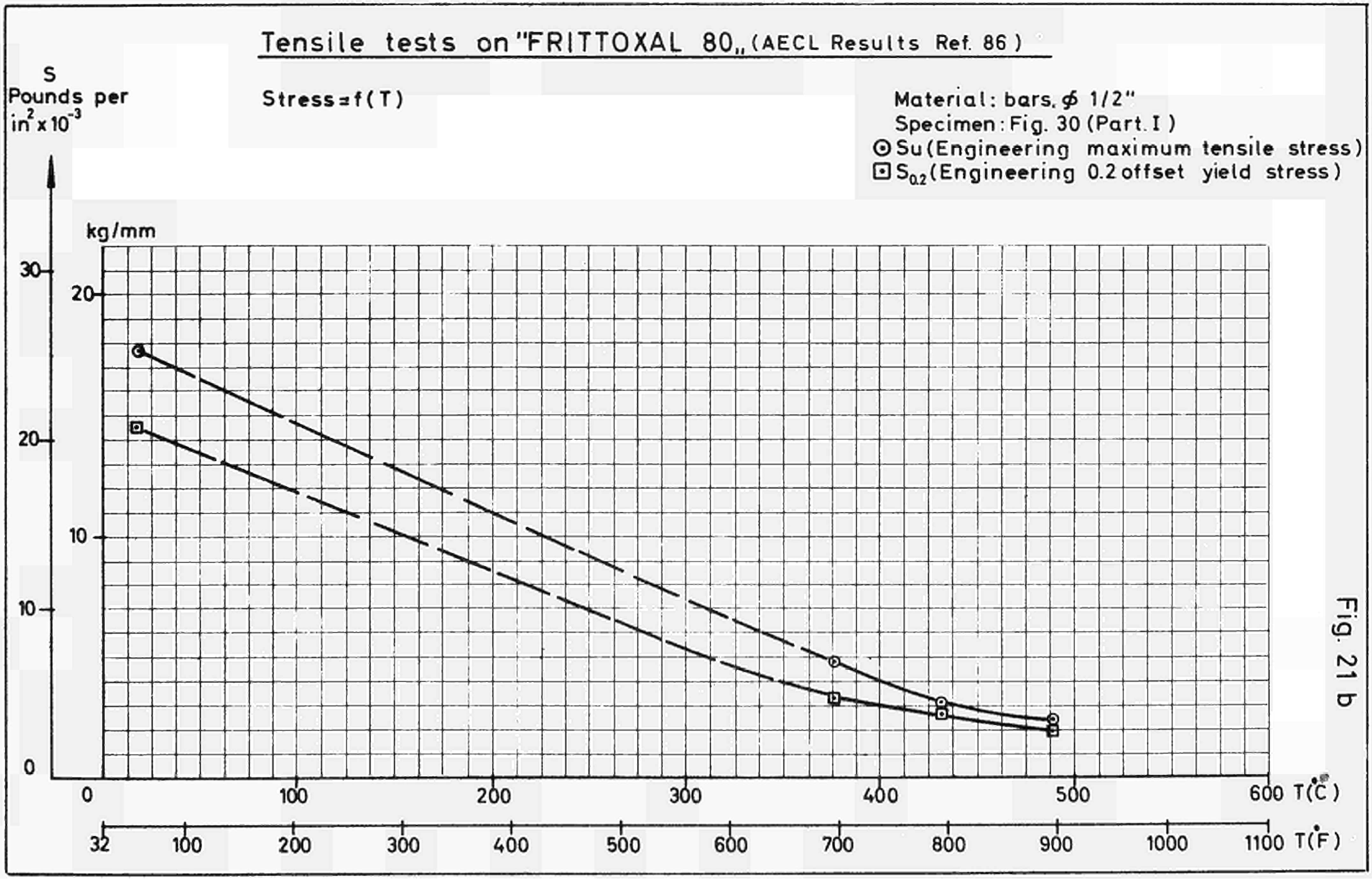

Tensile tests on "FRITTOXAL 80" (AECL Results Ref. 86)
Stress = f(T)
Material: bars, ϕ 1/2"
Specimen: Fig. 30 (Part. I)
Su (Engineering maximum tensile stress)
S0.2 (Engineering 0.2 offset yield stress)
S
Pounds per
in² x 10⁻³
kg/mm
30
20
10
0
20
10
0
100
200
300
400
500
600
T(°C)
32
100
200
300
400
500
600
700
800
900
1000
1100
T(°F)

Fig. 21 b

Tensile tests on "FRITTOXAL 20" (AECL Result Ref. 86)

Strain epb = f (T)

Material : bars ϕ 1/2"
Specimen : Fig. 30 (Part. I)
+ epb : (Engineering plastic strain at breaking point)

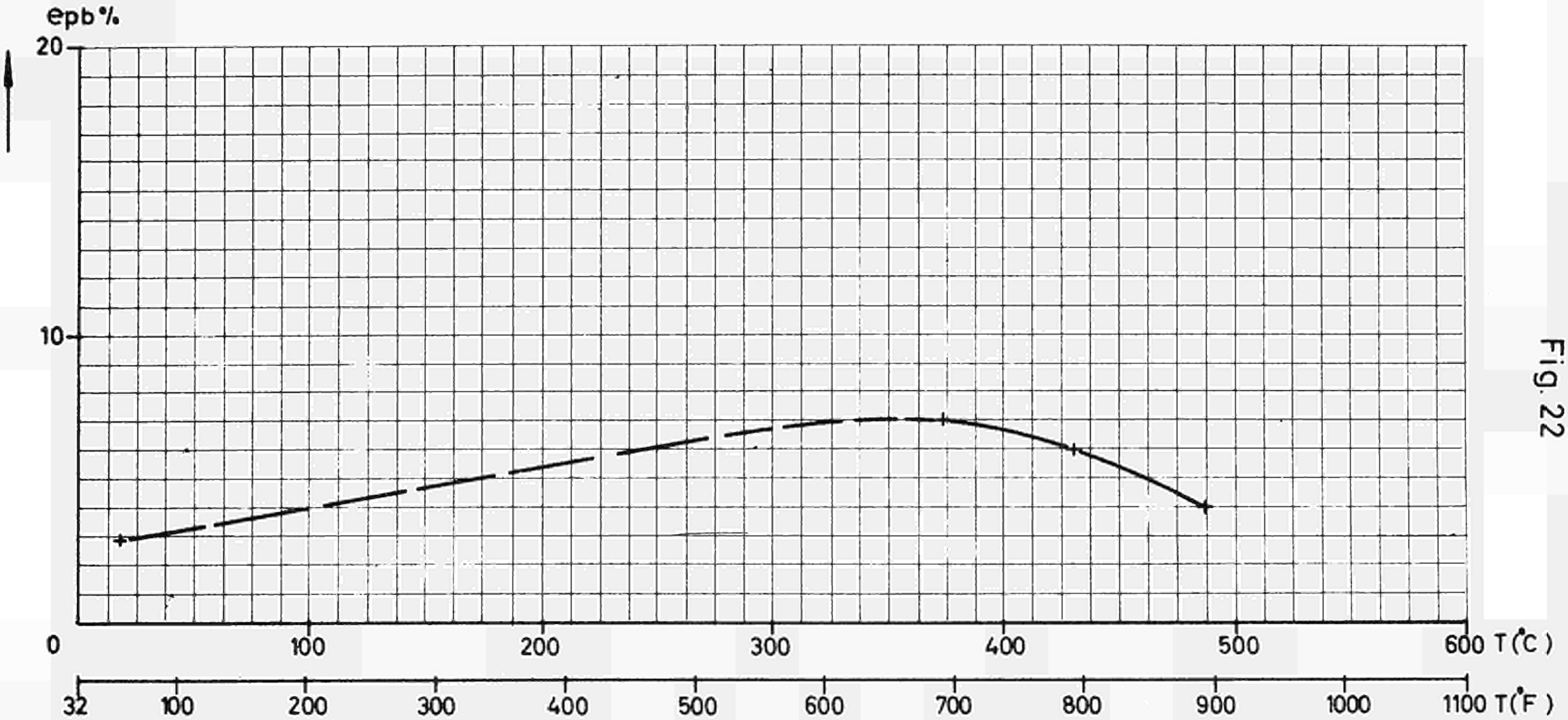


Fig. 22

Tensile tests on "FRITTOXAL 40" (AECL. Result Ref. 33-86)

Strain epb = f(T)

Material : bars ϕ 1/2"

Specimen : Fig. 30 (Part. I)

+epb : (Engineering plastic strain at breaking point)

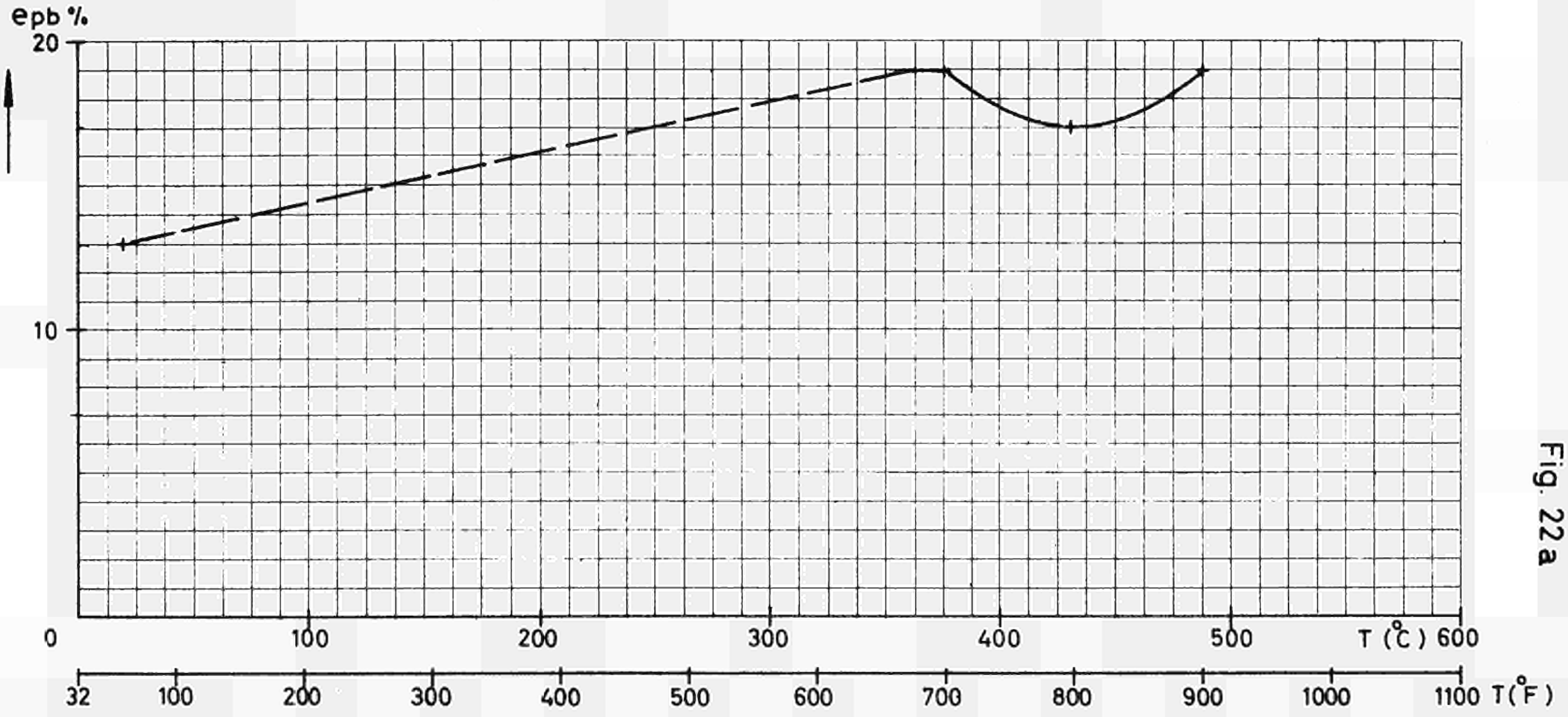


Fig. 22a

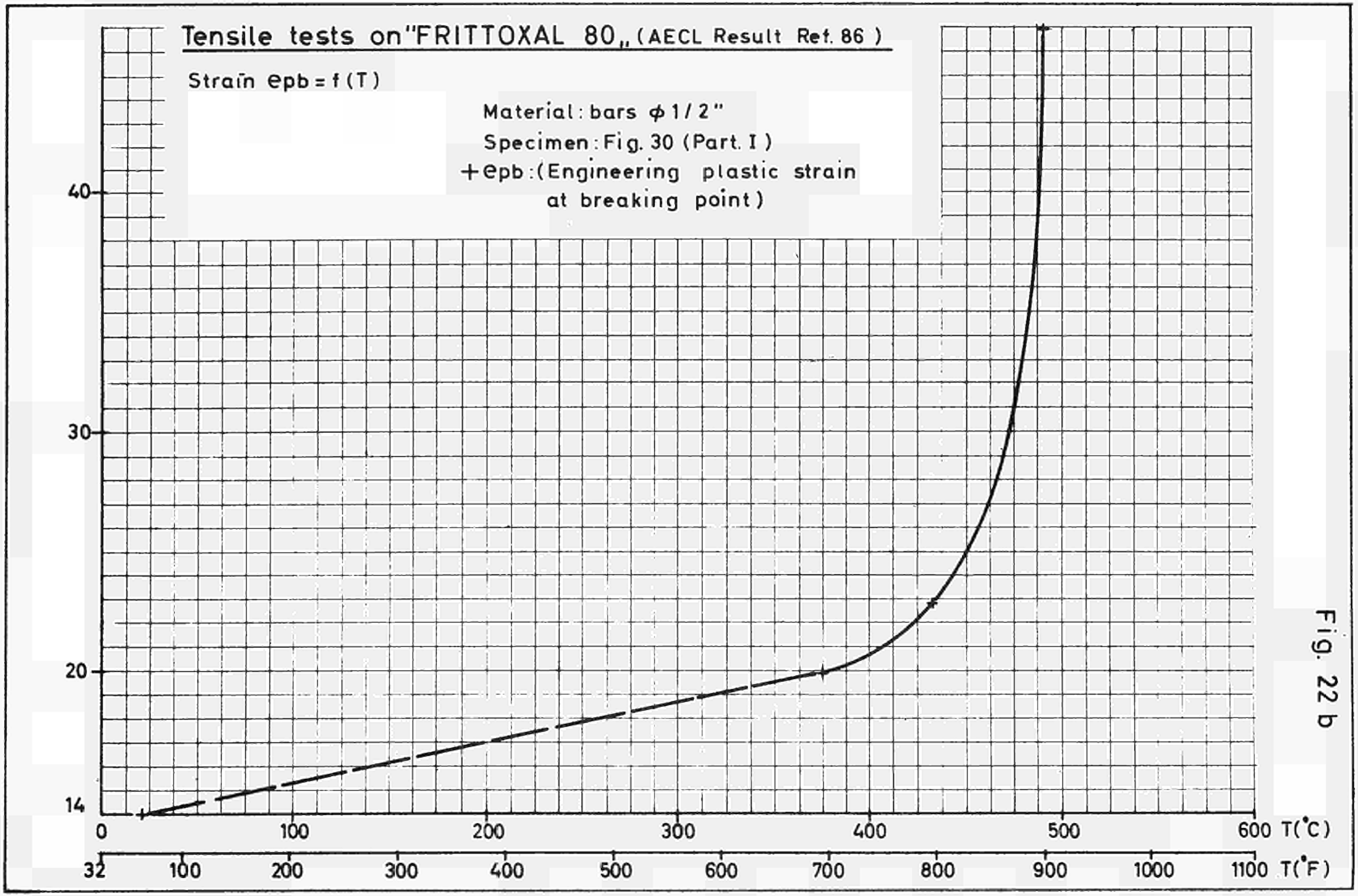

Tensile tests on "FRITTOXAL 80" (AECL Result Ref. 86)
Strain epb = f (T)
Material: bars φ 1/2"
Specimen: Fig. 30 (Part. I)
+ epb: (Engineering plastic strain at breaking point)
14
20
30
40
0
100
200
300
400
500
600
T(°C)
32
100
200
300
400
500
600
700
800
900
1000
1100
T(°F)

Fig. 22 b

Fig. 23

Histograms of mechanical Properties (Ref. 73.)

Su $S_{0.2}$ epb

Grade A-120-20-D   Grade A-50-10-D

20 °C   500 °C

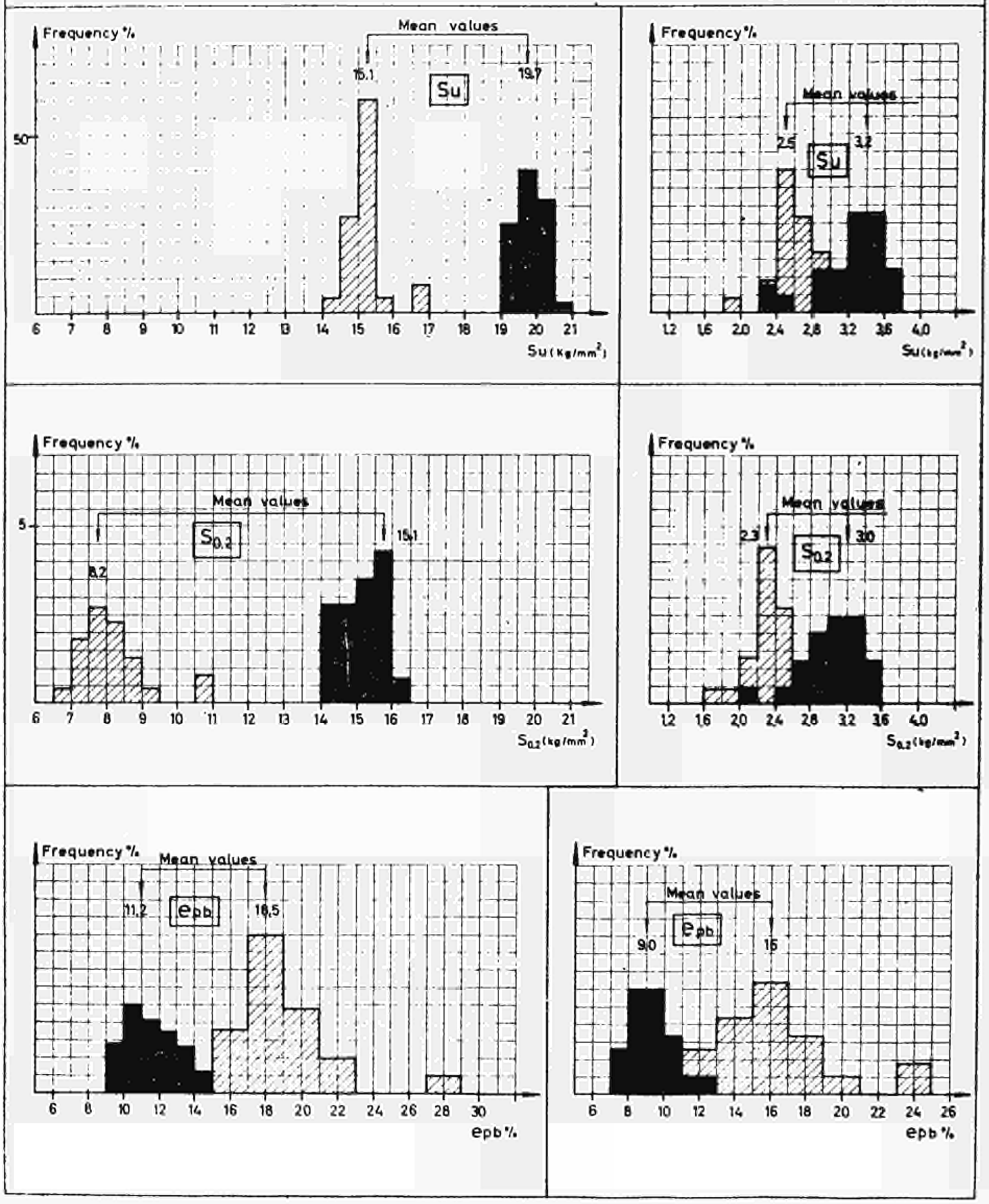


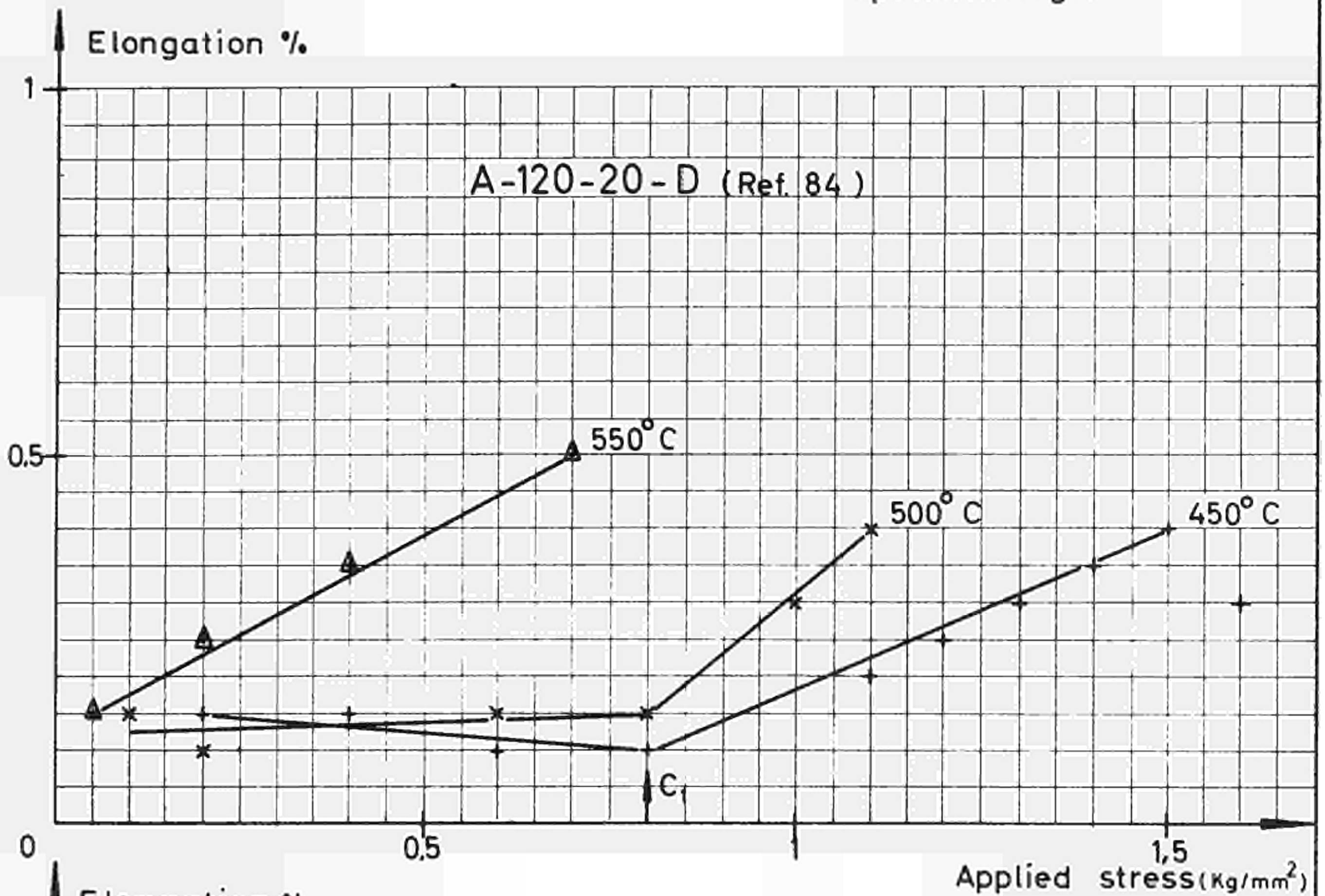
Fig. 24

Elongation after 100 H vs. applied stress

FRITTOXAL A-120-20-D A-50-10-D

Material: Tubes

Specimen: Fig: 7



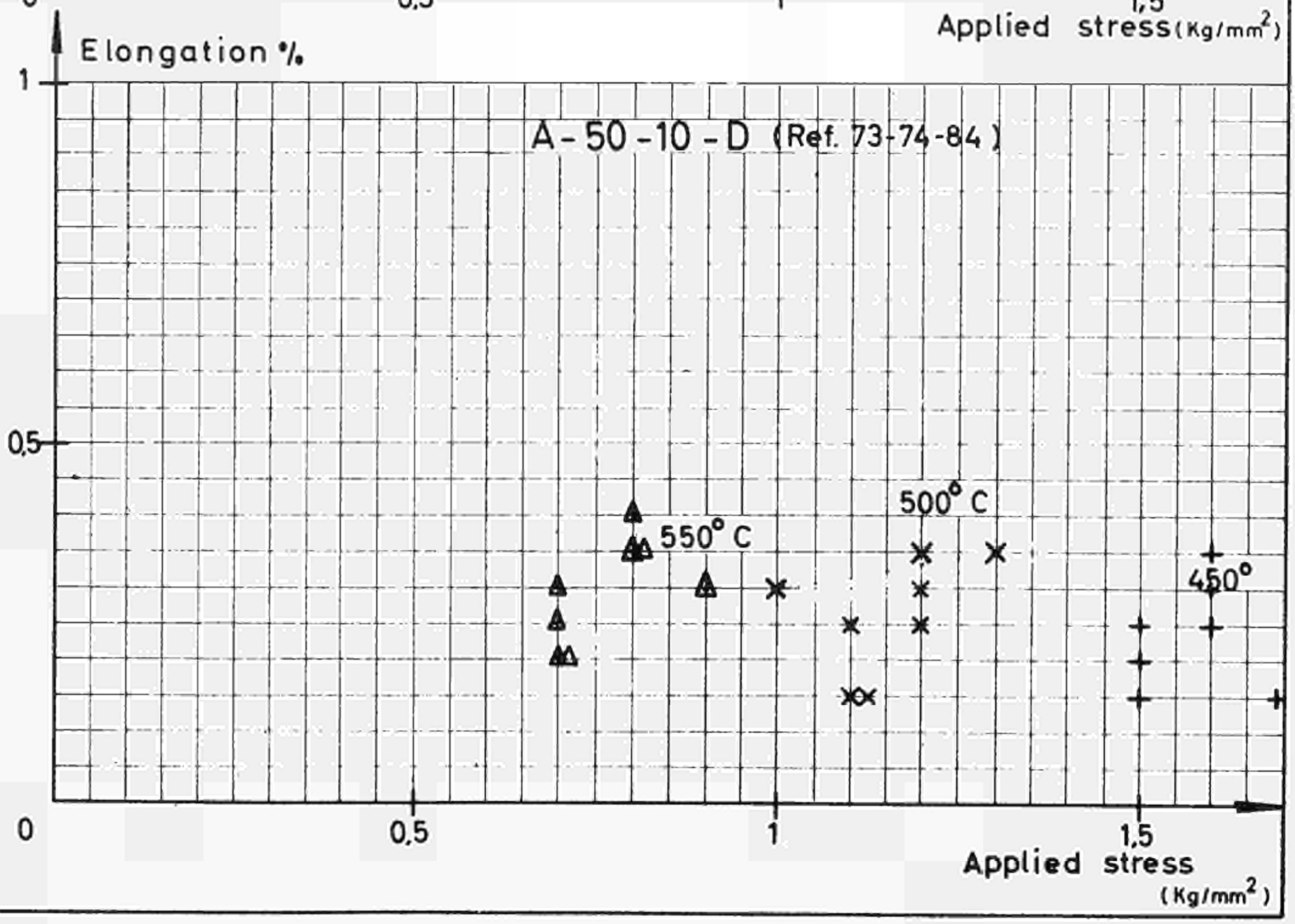
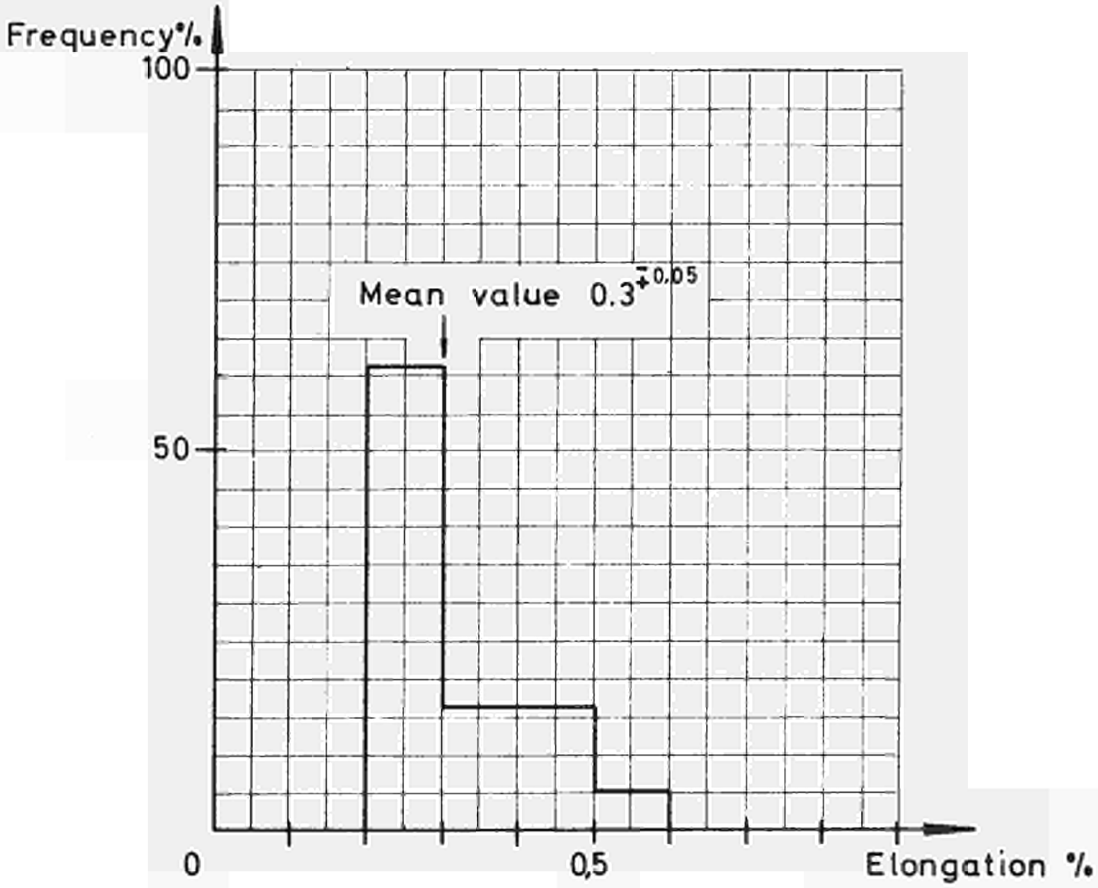


Fig. 25

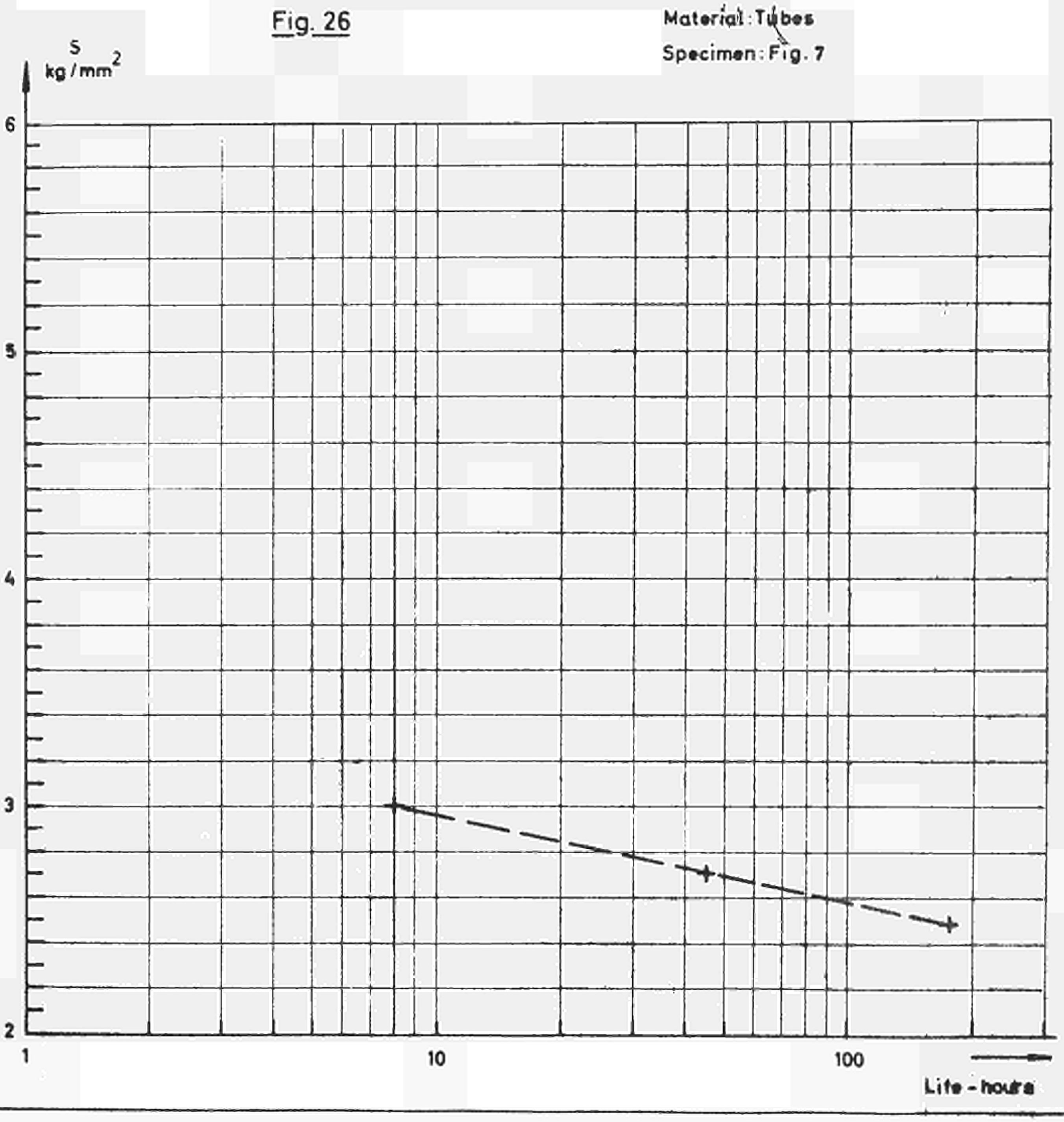
Histogram of elongation measured after 100^H at $T = 500\,^\circ C$ $\sigma = 1.1\ Kg/mm^2$ on FRITTOXAL A-120 - 20 - D (Ref. 73)

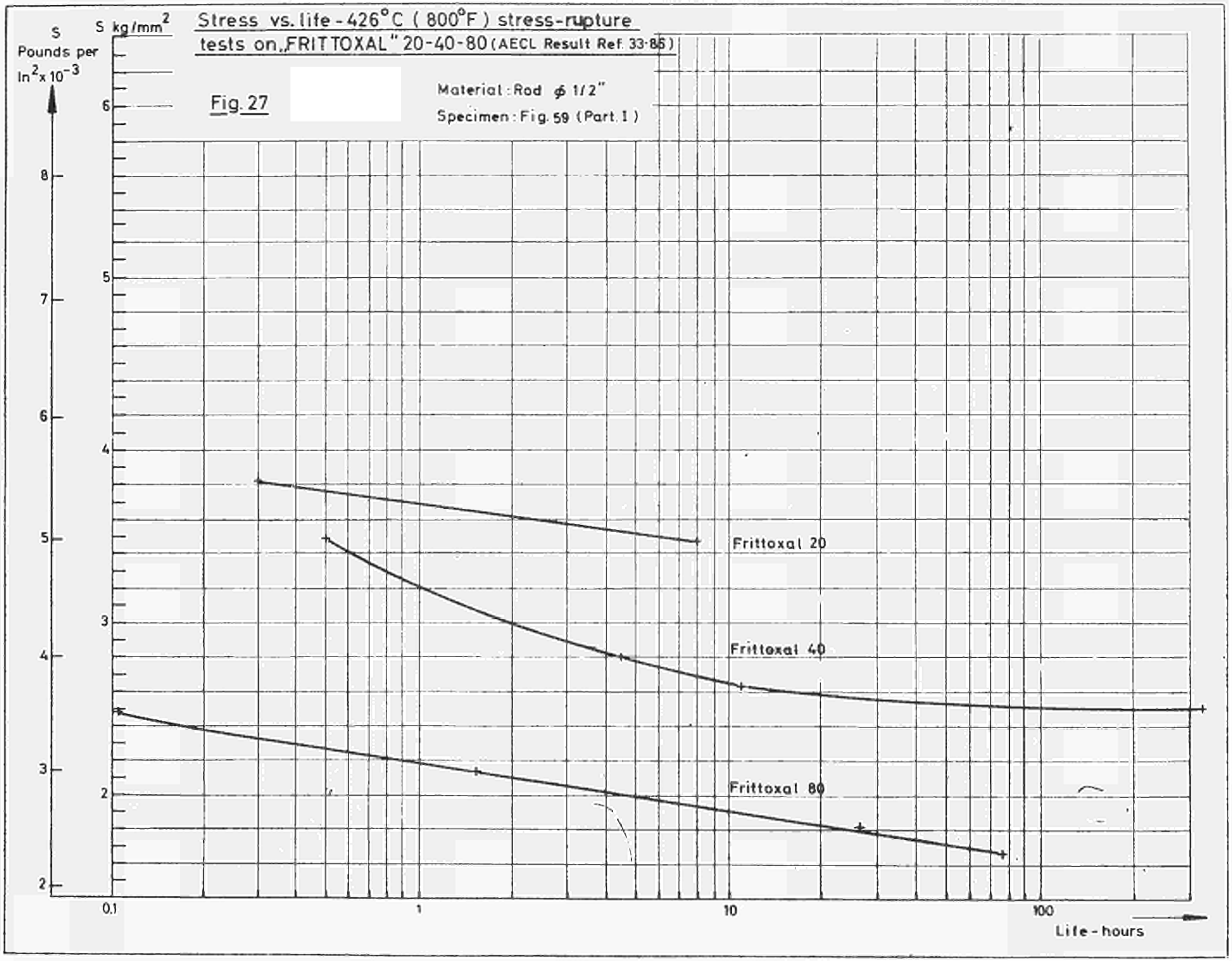
Material: Tubes (18 Results)
Specimen: Fig. 7



Stress vs. life stress-rupture tests on FRITTOXAL A-20-07-D at 450° C (Ref. 75)

Fig. 26





Stress vs. life - 426°C (800°F) stress-rupture tests on „FRITTOXAL" 20-40-80 (AECL Result Ref. 33-85)
Fig. 27
Material: Rod ϕ 1/2"
Specimen: Fig. 59 (Part. I)
S Pounds per In²x 10⁻³
S kg/mm²
Frittoxal 20
Frittoxal 40
Frittoxal 80
Life - hours
0.1
1
10
100

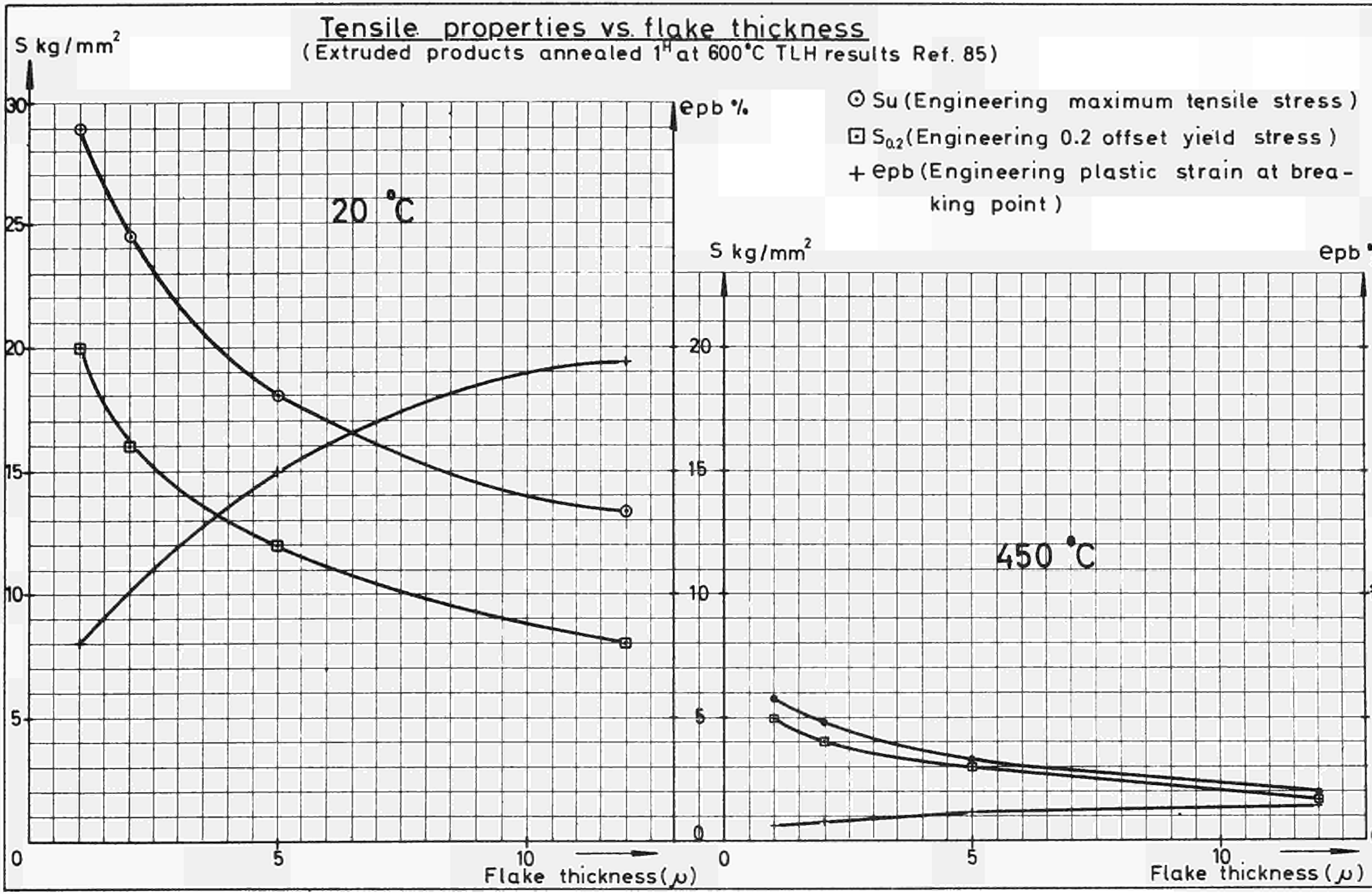
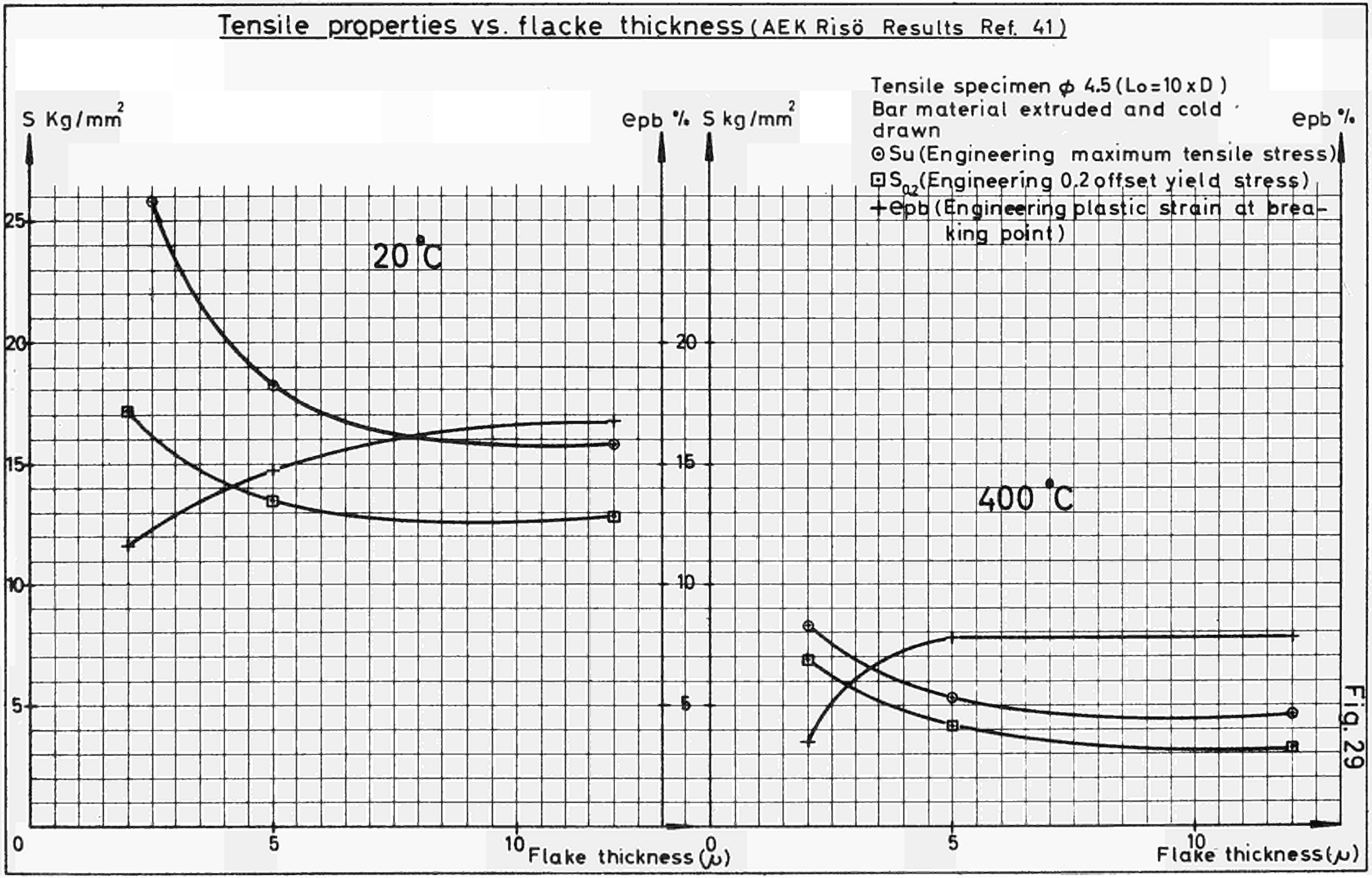

Tensile properties vs. flake thickness
(Extruded products annealed 1H at 600°C TLH results Ref. 85)
⊙ Su (Engineering maximum tensile stress)
⊡ S0.2 (Engineering 0.2 offset yield stress)
+ epb (Engineering plastic strain at breaking point)
S kg/mm²
epb %
20 °C
450 °C
Flake thickness (μ)
Flake thickness (μ)
0
5
10
15
20
25
30

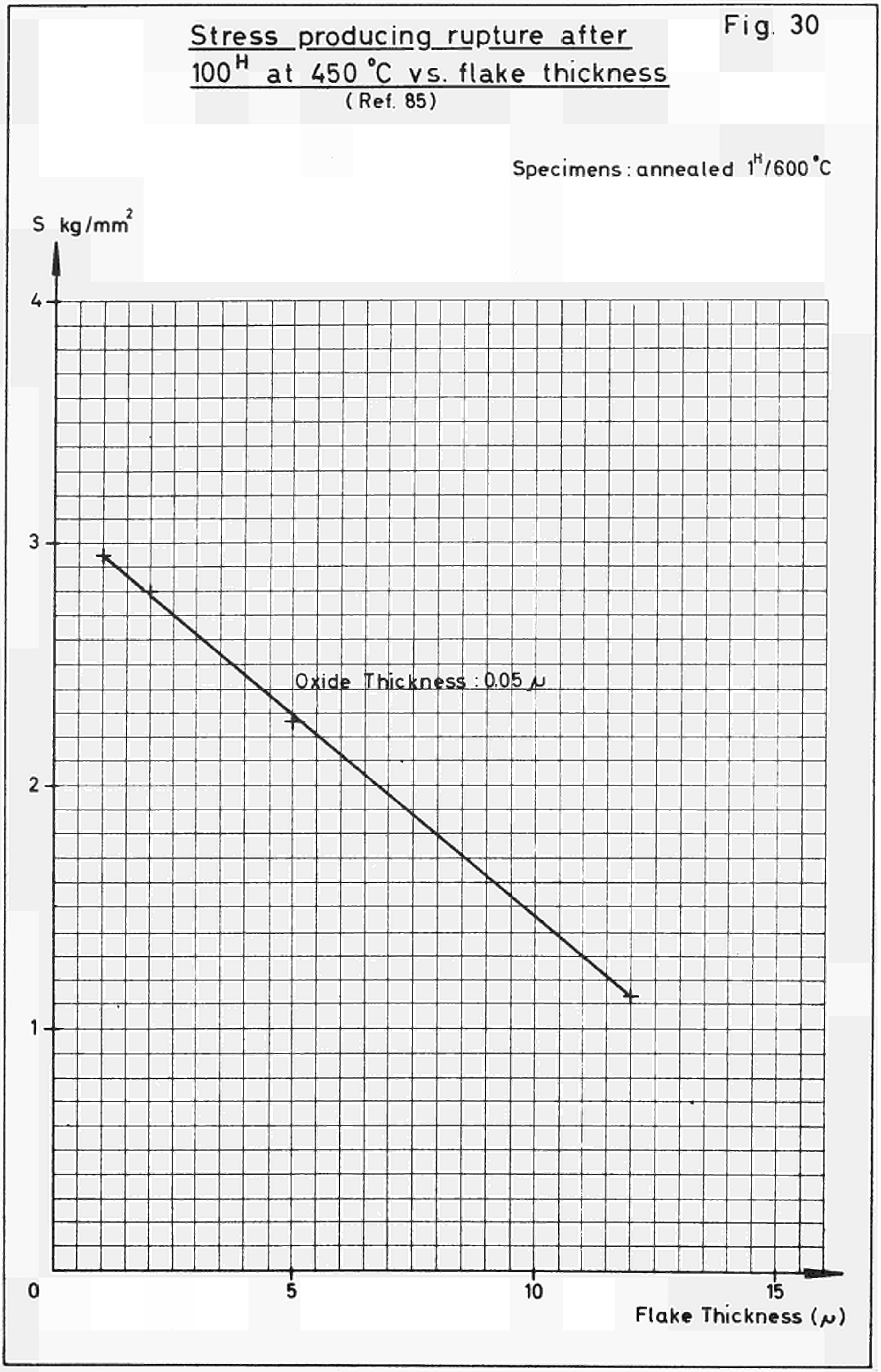
Fig. 28



Tensile properties vs. flacke thickness (AEK Risö Results Ref. 41)
Tensile specimen ϕ 4.5 (Lo=10 x D)
Bar material extruded and cold drawn
⊙ Su (Engineering maximum tensile stress)
⊡ S0.2 (Engineering 0.2 offset yield stress)
+ epb (Engineering plastic strain at breaking point)
S Kg/mm2
epb %
S kg/mm2
epb %
20 °C
400 °C
25
20
15
10
5
0
5
10
Flake thickness (μ)
0
5
10
Flake thickness (μ)

Fig. 29

Fig. 30



Stress producing rupture after
100 H at 450 °C vs. flake thickness
(Ref. 85)
Specimens : annealed 1 H/600 °C
S kg/mm²
4
3
2
1
0
5
10
15
Flake Thickness (μ)
Oxide Thickness : 0.05 μ

Fig. 31

Tensile properties vs. oxide thickness (Ref. 85)

(Extruded products annealed 1^{H} at 600 °C)

⊙ Su (Engineering maximum tensile stress)
⊡ $S_{0.2}$ (Engineering 0.2 offset yield stress)
+ epb (Engineering plastic strain at breaking point)

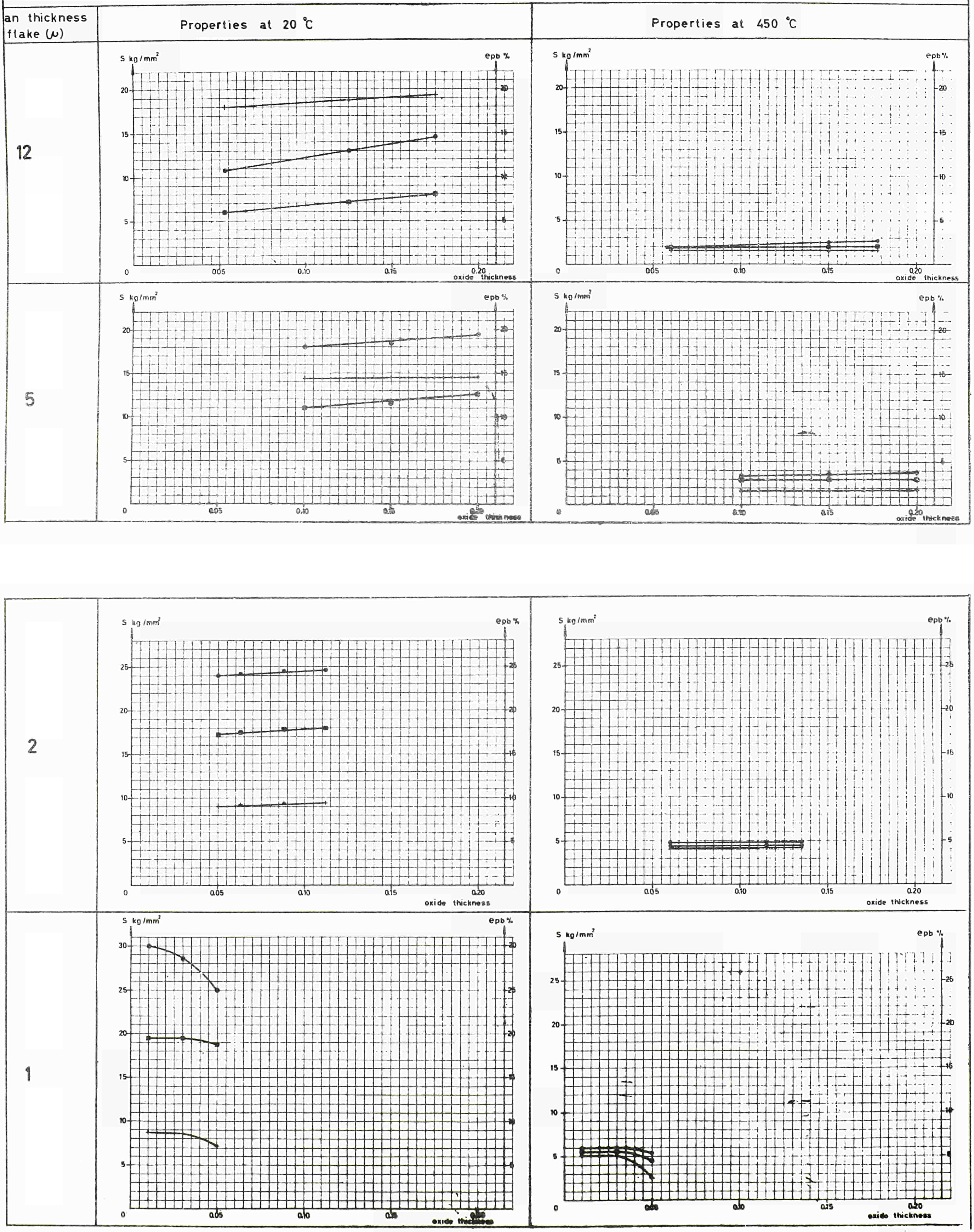
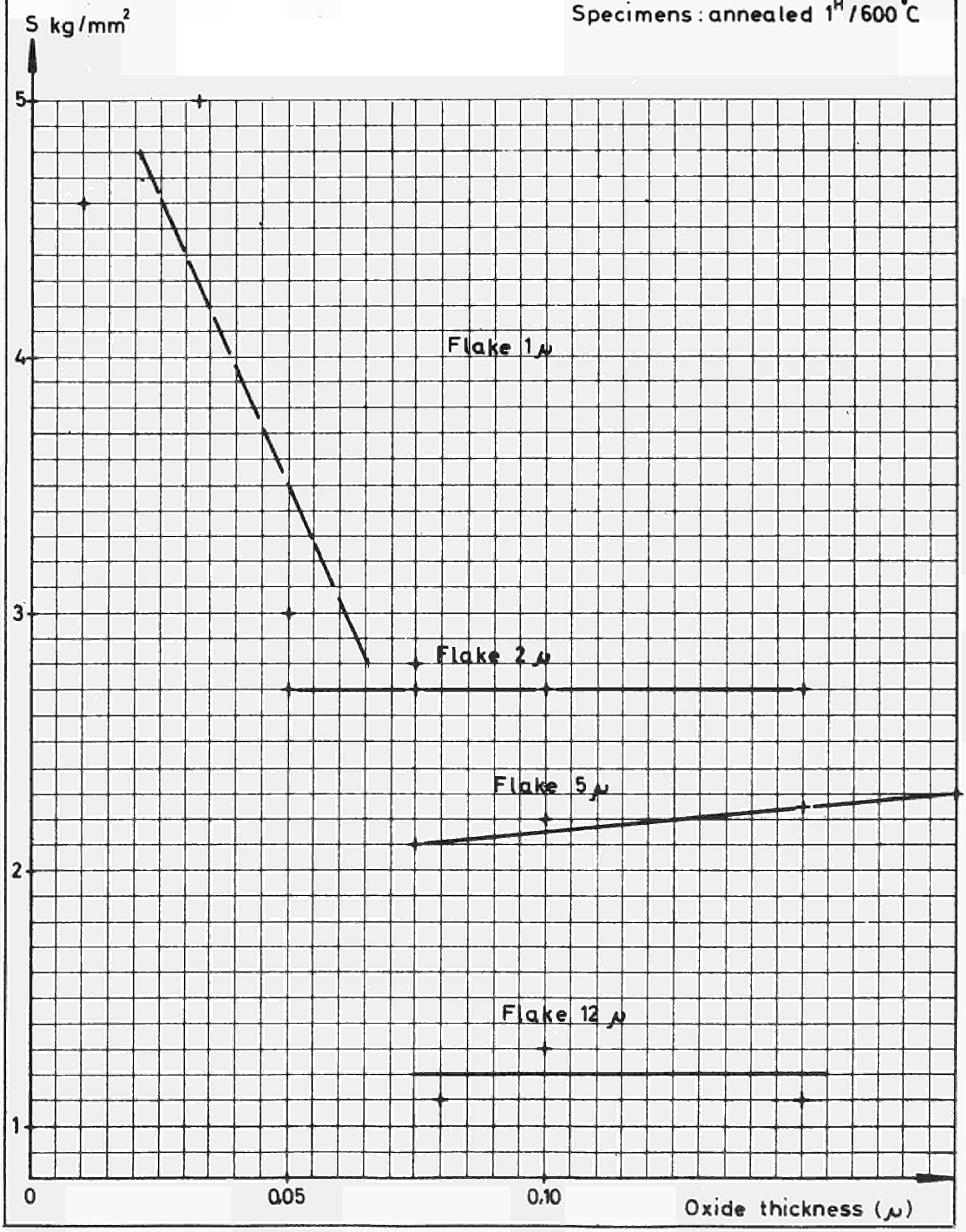
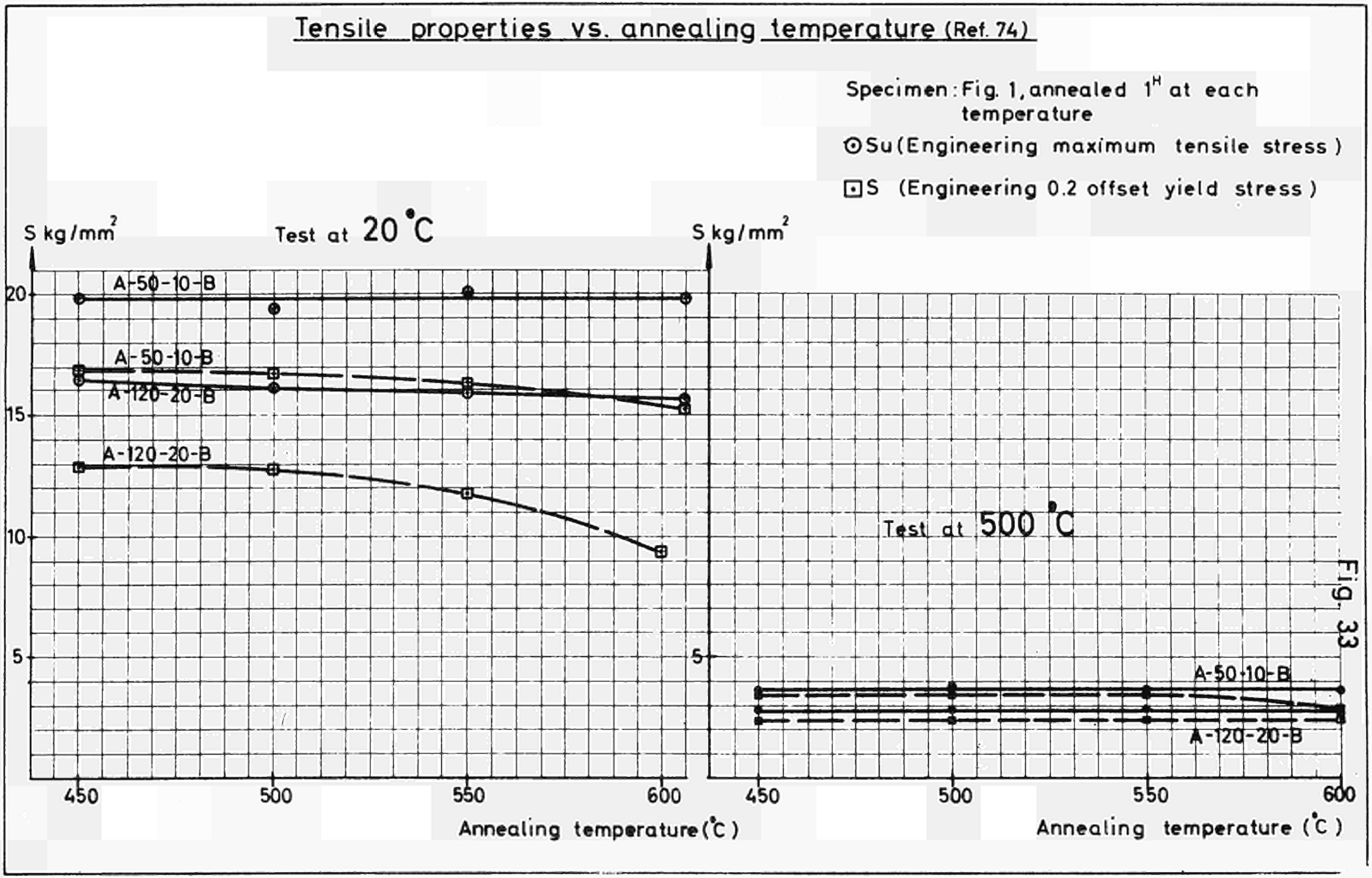


Fig. 32

Stress producing rupture after 100^{H} at 450 °C vs. oxide thickness

(Ref. 82-85)





Tensile properties vs. annealing temperature (Ref. 74)
Specimen: Fig. 1, annealed 1H at each temperature
⊙ Su (Engineering maximum tensile stress)
⊡ S (Engineering 0.2 offset yield stress)
S kg/mm²
Test at 20 °C
S kg/mm²
A-50-10-B
A-50-10-B
A-120-20-B
A-120-20-B
20
15
10
5
Test at 500 °C
5
A-50-10-B
A-120-20-B
450
500
550
600
450
500
550
600
Annealing temperature (°C)
Annealing temperature (°C)

Fig. 33

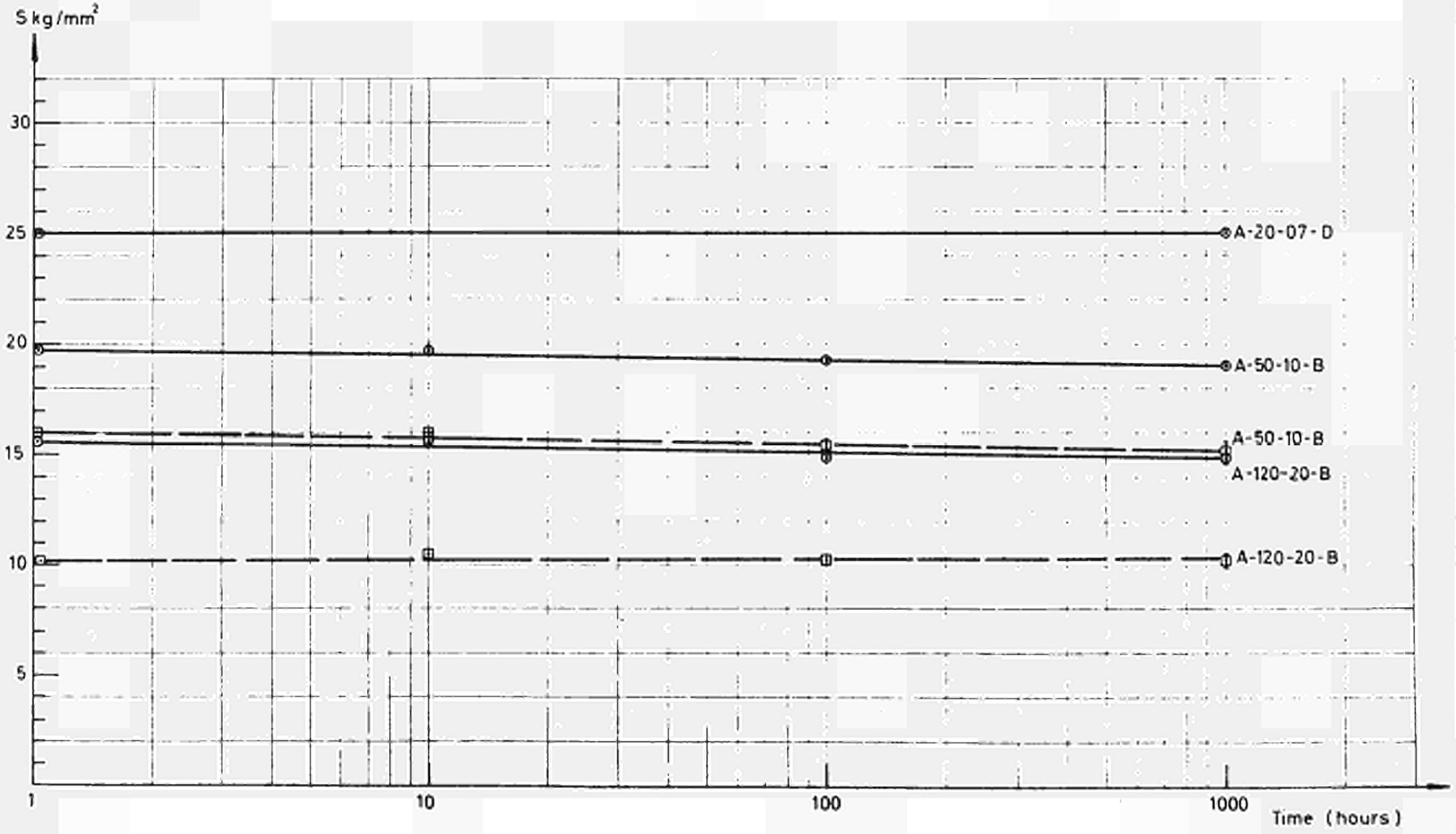
Tensile properties vs. annealing time at 20 °C annealing temperature 450° (Ref. 76)

Specimen: Fig. 1

⊙ Su (Engineering maximum tensile stress)

⊡ $S_{0.2}$ (Engineering 0.2 offset yield stress)

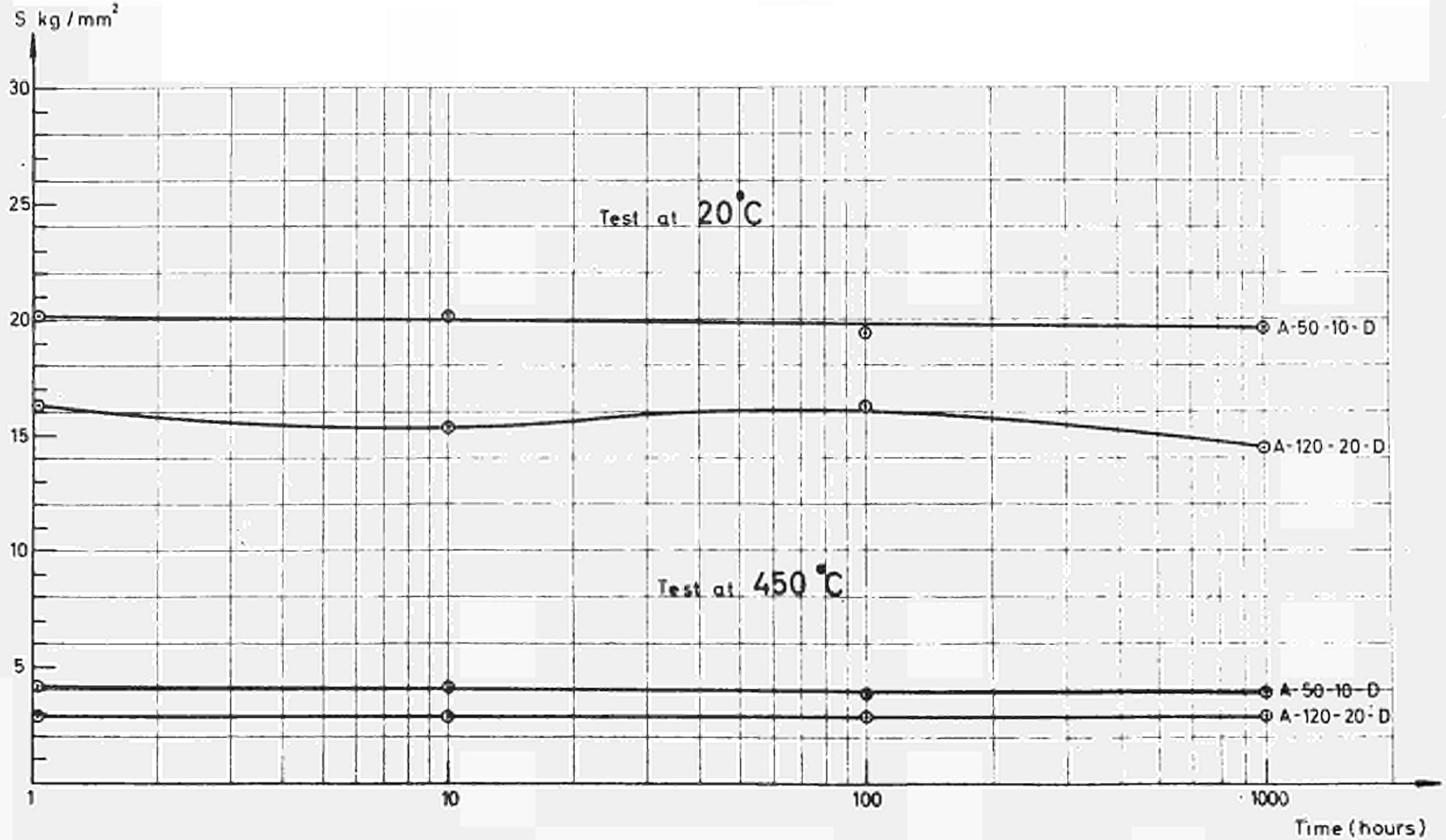
Fig. 34



Tensile properties vs. annealing time at 20 °C and 450 °C (annealing temperature : 600 °C)

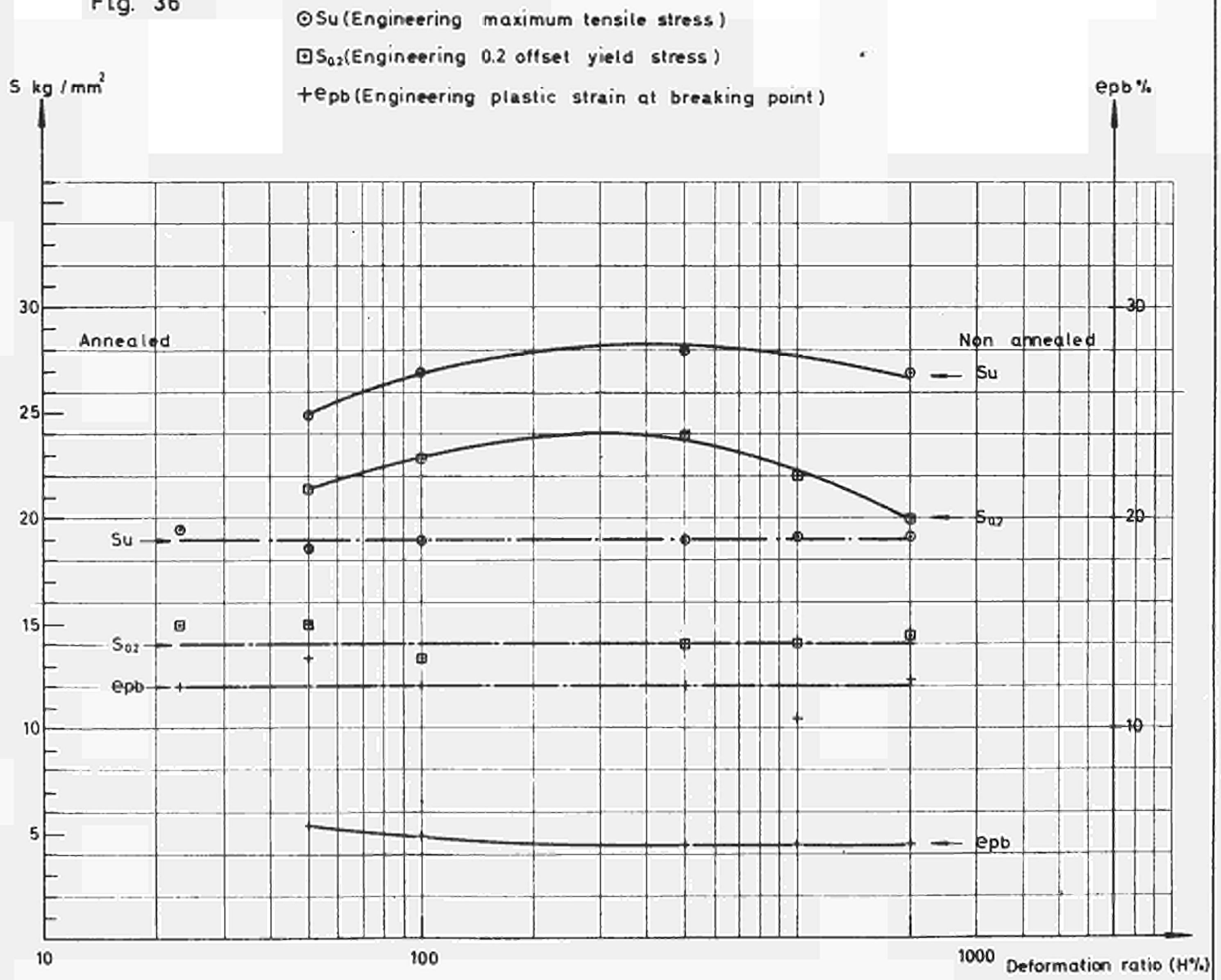
(Ref. 77)

Fig. 35



Tensile properties at 20 °C of "FRITTOXAL" A-50-10-D vs. deformation ratio (Ref. 85)

Fig. 36



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To disseminate knowledge is to disseminate prosperity — I mean general prosperity and not individual riches — and with prosperity disappears the greater part of the evil which is our heritage from darker times.

Alfred Nobel

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